

October 14, 2015  
File No. 01.0024065.16

Ms. Gloria Toro  
Puerto Rico Environmental Quality Board  
Land Pollution Branch  
Cruz A. Matos Environmental Agencies Building  
1375 Ponce de Leon Ave.  
San Juan, Puerto Rico 00926-2604



Re: Revised Intrinsic Biodegradation Study Work Plan – Revision 1  
Hewlett-Packard Company Voluntary Remediation Project  
San German, Puerto Rico

249 Vanderbilt Ave.,  
Norwood,  
Massachusetts  
02062  
781-278-3700  
FAX 781-278-5701  
<http://www.gza.com>

Dear Ms. Toro:

GZA GeoEnvironmental, Inc. (GZA), on behalf of Hewlett-Packard Company, is pleased to submit to the Puerto Rico Environmental Quality Board the enclosed revised first revision to the work plan for evaluating intrinsic biodegradation and an Enhanced Reductive Dechlorination (ERD) pilot test at the above referenced location (the "Site"). Revision of the original IB Work Plan was requested by PREQB in its letter approving the installation of additional on- and off-Site wells dated November 22, 2013. GZA issued a revised work plan (Intrinsic Biodegradation Study Work Plan – Revision 1) to PREQB on April 6, 2015 and on July 2, 2015, PREQB provided their comments. Response to PREQB's July 2015 comments are attached and applicable edits have been incorporated into this revised work plan.

Appended to this IB Work Plan is a revised Quality Assurance Project Plan (QAPP) that also incorporates applicable edits based on PREQB's July 2015 comments. The QAPP will provide guidance on data collection to properly evaluate intrinsic biodegradation and the ERD pilot test.

We, respectfully, request your approval of this revised work plan and QAPP. Please contact the undersigned or Mr. Paul Paschke (Hewlett-Packard Company) at 970-898-0573 or paul.paschke@hp.com, if you have any questions.

Very truly yours,

GZA GEOENVIRONMENTAL, INC.

A handwritten signature in blue ink, appearing to read "John A. Colbert".

John A. Colbert  
Senior Project Manager

A handwritten signature in blue ink, appearing to read "Charles A. Lindberg".

Charles A. Lindberg  
Senior Principal

A handwritten signature in blue ink, appearing to read "Karen Kinsella".

Karen Kinsella  
Consultant/Reviewer

Attachments: GZA's Response to PREQB's Comments  
Revised Intrinsic Biodegradation Study Work Plan – Revision 1 (October 2015)

cc: File  
Díaz, Lorena Rodríguez; Puerto Rico Environmental Quality Board  
Roman, Frances M. Segarra; Puerto Rico Environmental Quality Board  
Aviles, Jesse; United States Environmental Protection Agency Region II  
Morales, Jorge; Puerto Rico Industrial Development Company  
Meléndez, Joel; Puerto Rico Industrial Development Company  
Fornés, Karen; Puerto Rico Industrial Development Company  
Paschke, Paul; Hewlett-Packard Company



**GZA's Response to PREQB's Comments**  
on  
**Intrinsic Biodegradation Study Work Plan – Revision 1**  
**Voluntary Remediation Project**  
**Hewlett-Packard Company (HP)**  
**San German, Puerto Rico**  
**PRD-991291857**  
prepared by  
**GZA GeoEnvironmental, Inc., of**  
**Norwood, Massachusetts**

Dated: October 14, 2015

**The following comments are from a Review of Intrinsic Biodegradation Study Work Plan – Revision 1 from Weldin F. Ortiz Franco, Executive Director of the LPCA division of Puerto Rico Environmental Quality Board (PREQB) dated July 2, 2015.**

**Work Plan Comments**

1. Page 1, Section 2, First Paragraph: The document text refers the reader to the Intrinsic Bioremediation Study Work Plan submitted in May 14, 2010 for additional background and description to the site. Although the regulatory agencies have electronic and hard copy of the IBS Work Plan (May 2010), in order to accept that the background and description of the site is not included in this revision of the document, it should be included as an appendix, to provide the reader a standalone document. It is PREQB's recommendation to include the IBS Work Plan text as Appendix C to the current revision.

*The revised Intrinsic Bioremediation Study Work Plan submitted on September 27, 2010 has been added as Appendix A. This is the latest version of original IBS Work Plan and the reference to the document has been updated.*

2. Page 5, Section 3.5:
  - a. Please include the Safety Data Sheets (SDS) for the proposed substrate to inject (Anaerobic Biochem (ABC®)).

*Safety Data Sheets for both proposed substrate Anaerobic Biochem (ABC®) and the proposed bacterial mixture KB-1® have been added as Appendix C.*

- b. It is establish that the goal of the Enhanced Reductive Dechlorination (ERD) pilot text is to inject as much carbon as practical unto the saprolite fill. At least the explanation of the calculations to determine how much will be practical should be included.

*Based on previous constant head tests performed at OW-101 and OW-307, measured hydraulic conductivity appears to be between 0.1 and 1.0 feet/day. Based on these values and previous pumping tests performed at the Site, we anticipate being able to gravity inject at an average rate of approximately one gallon per minute. If conditions are encountered which*

*indicate that the actual injection rate is significantly different than the estimated rate, the concentrations of ABC additive will be adjusted accordingly to maximize the quantity of additive injected and the volume of the treated aquifer. That is, a more dilute or more concentrated solution of ABC additive will be injected. The mass of ABC injected will be the same.*



3. Page 5, Section 4.2, Performance Monitoring: As it is written the text gave the wrong impression that all the wells screened in the alluvium/fill, saprolite and bedrock will be sample for all the parameters. A reference to Worksheet #18 of the Quality Assurance Project Plan (QAPP) should be included.

*The text has been modified to note that only a subset of wells screened across the alluvium/fill, saprolite, and bedrock will be analyzed for biodegradation screening parameters. A reference to Worksheet #18 of the Quality Assurance Project Plan (QAPP) which describes the sampling locations and analytical methods has been added.*

4. Page 6, Section 4.3, Sampling Parameters: For clarity, the list of the cVOCs concentrations that are going to be measured should be included in this section or at least a reference to were in the document can be encountered.

*The text has been modified to list each cVOC analyzed under the modified 8260 list for the Site.*

5. Page 7, Section 5.0, Reporting: Semi-Annual status reports are encouraged to provide follow up on status of the cleanup, however, once the IBS is considered finished, an IBS Report should be prepared with conclusions and recommendations. This report should be considered in this section too.

*At this time, we do not believe an IBS completion report is possible. Please see reasoning below, in response to Comment #6.*

6. In general, it is PREQB recommendation to add a Section to discuss an Exit Strategy, what parameters at which levels will deem the Study to prove that IB is occurring and is a suitable remedy for the site. Although Worksheet #10 of the QAPP mentioned it, at this section, what is the expected time needed to finish the IBS should be included.

*At this time, it is unrealistic to anticipate a time to finish the IBS. The ERD pilot test described in the IB Work Plan is designed to investigate whether additive injections can enhance and accelerate IB. It may take several years to evaluate whether the ERD pilot injections have effectively accelerated IB. At that time, it is more likely that we will be able to anticipate a time frame for the IBS completion report.*

#### Quality Assurance Project Plan Comments:

7. Worksheet #1: The PREQB's Project Managers are not authorize to provide signature approval to any document. However, the PREQB's Approval or No Further Comment letter signed by the nominated authority should serve as approval to the document.

Therefore it is recommended to delete Gloria Toro Agrait name from the Title and Approval Page.

*Gloria Toro Agrait's name has been deleted from the Title and Approval Page.*



8. Worksheet #2: In item 3.5 the crosswalk provided for a Data Management SOPs is Appendix C of the QAPP, it should be noticed that Appendix C contains a General Guideline for Quality Control instead of a Data Management SOP.

*Text has been revised as noted above.*

9. Worksheet #3: The correct PREQB's Land Pollution Control Area fax number is 787-767-8118.

*The text has been revised to reflect the correct fax number.*

10. Worksheet #6: Please add extension 3586 to the Gloria Toro Agrait phone number.

*The text has been revised to add extension 3586 to Gloria Toro Agrait's phone number.*

11. Worksheet #11: Please specify how it would be determined that the project have a "sufficient number of sample" to evaluate the efficacy of the remedial actions.

*Groundwater monitoring focuses on sampling areas with elevated cVOCs, as well as groundwater downgradient of the higher cVOC concentrations and off-Site groundwater sentinel wells. Because IB is a very slow process, we believe the current sampling frequency is sufficient to evaluate the extent of groundwater impacts and associated risks, and efficacy of the remedial approach.*

12. Worksheet #14: Clarification is needed regarding if the reference to Worksheet #13 for secondary data is being made towards secondary lines of evidence.

*The reference in Worksheet #14 is referencing both groundwater data and biodegradation parameter data. GZA will provide further clarification, if needed.*

13. Worksheet #18:

- a. For the parameters that would be sampled semi-annually for cVOCs and IB Indicators (first line of the table), please notice that according to Table 1 of the IBS Work Plan and first column of this worksheet there are 14 wells to be sampled, however the number of samples column (sixth column) indicates only 11 field samples. Please revise the document accordingly.

*The "number of samples" column has been revised to indicate that 14 field samples will be collected.*





- b. Table 1 does not include TOC as a field parameter and the QAPP indicate that TOC will be analyzed in the laboratory. Please revise the document accordingly.

*TOC has been removed as a field parameter.*

- 14. Worksheet #20:
  - a. Please add the superscript 2 to the Total No. of Samples to Lab's column (last column).

*Text has been revised as noted above.*

- b. Please add a note to the table to indicate that the SOPs were included on the QAPP's Appendix A. Also, please clarify where the reference number for the Analytical Method's SOP can be found.

*Text has been revised as noted above. The method reference numbers are located in QAPP Worksheet #23: Analytical SOP References Table.*

- 15. Worksheet #22: It is not clear what does the number after the slash means under the SOP Reference. Using that number the SOP could not be found.

*The slash and number have been removed from the SOP reference.*

- 16. Worksheet #23: Please add a note indicating where the SOPs can be found.

*A footnote has been added to worksheet #23 noting that the laboratory SOPs can be found in Appendix A of the QAPP.*

- 17. Worksheet #28: According to Table 1, the number of sampling locations are 57, according to Worksheet #18 there are 54 sampling locations and this worksheet mentioned 55. Please clarify and revise accordingly.

*Worksheet #18 and Worksheet #28 have been revised to indicate 57 sampling locations.*

- 18. Appendix C: Please explain why broken bottles were not included as one of the situations where the Laboratory should contact GZA project manager or QA Officer before proceeding to the analysis.

*Appendix C has been revised to include broken sample bottles as a situation where the Laboratory should contact the GZA project manager or QA Officer before proceeding to the analysis.*

- 19. Appendix D:
  - a. SOP No. 2.4, Bullet 2.b: Reference to A.2 is made in order to discontinue to intermittently surge and overpump the well. Please clarify since no A.2 was found in the SOPs.

*This has been revised to reference 2.a.*



b. SOP No. 3.1.2:

- i. Equipment and Materials: For the sample containers reference is made to attached Table 1. It was not found, please clarify. There are other references to Table 1 through the SOP.

*References to Table 1 have been removed from the SOP.*

- ii. Procedures: It is established that well sampling sequence would be based on previous analytical data, if available. Obviously for the majority of the wells there is previous analytical data available. This sequence should be discussed and justified at least in the semi-annual reports.

*When possible, wells will be sampled in sequence from lowest concentration to highest concentration. Dedicated tubing at all locations and dedicated pumps will be used when practical. Water level meters and non-dedicated pumps will be decontaminated between each sampling location withalconox and deionized water.*

20. Appendix E: This appendix should include the equipment user manuals, however, for the Proactive Pump and Controller an user manual was not included, only a brochure with general information of the features were included.

*The equipment manufacturer indicated that a user manual has not been created for the Proactive Pump and Controller due to the simplicity of the pump and the controller.*

Quality Assurance and Quality Control Office's Comments

A. UFP-QAPP Worksheet #1 (s 1-2) (Title and Approval):

- a. Comment A-1: Although in 2 this QAPP the PREQB Hazardous Waste Permit Division Project Manager was included in the Approval Sheet, the PREQB QA/QC Specialist Manager (QAO) was not. Currently, the PREQB Hazardous Waste Program Quality Management Plan establishes, as part of the Systematic Planning process, the role of the QAO in the technical and quality document review of technical documents related to RCRA corrective Action and Permitting activities, such as QAPP.
- b. Recommendation A-1: In order to be consistent with the Systematic Planning Process being implemented by the PREQB Land Pollution Control Area - Hazardous Waste Program (HWP) and recommendations made from the reviews of other QAPPs process by the QAO, it is recommended that the name of PREQB QA/QC Specialist Manager, Mrs. Frances M. Segarra Roman, be added as one of the approval signatures.

*Text has been revised as noted above.*



B. UFP-QAPP Worksheet # 3 ( 7) (Distribution List):

- a. Comment B-1: The PREQB QA/QC Specialist Manager (QA/QCM), Mrs. Frances M. Segarra Roman, was also not included in the Distribution List included in this worksheets; even though, as part of the Systematic Planning Process being implemented by the PREQB Land Pollution Control Area - Hazardous Waste Program (HWP), the QA/QCM may be performing data quality assessments and verification, and field technical audits. All based upon the requirements established in the QAPPs.
- b. Recommendation B-1: Recommend that an additional row be incorporated to include the QA/QCM as one of the QAPPs recipient, with the following information:

QAPP Recipient:	Frances M. Segarra Roman
Title:	QA/QC Specialist Manager
Organization:	EQB
Telephone:	787-767-8181, Ext. 3575
Fax Number:	787-767-8118;
E-mail Address:	francessegarra@j ca.pr.gov

Summary of the QA/QC Specialist Manager Project Oversight Activities:

Provide support to the HWPD in the quality and technical review of Corrective Actions Work Plans, RFI Sampling Analysis Plans, QA/QC data reports submitted for Corrective Actions investigations, QAPP for investigative or remedial project, etc. Performs technical and quality document review and field oversight/auditing activities as is part of the Systematic Planning Process being implemented as established by the PREQB LPCA Quality Management Plan.

*Text has been revised as noted above.*

C. UFP-QAPP Worksheet # 5 ( 10) (Project Organizational Chart):

- a. Comment C-1: See Comments A-1 and B-1.
- b. Recommendation C-1: Recommend to include the QA/QCM in the same box of the Project Organizational Chart where Mrs. Gloria M. Toro Agrait, EQB Project Manager, was included.

QAPP Recipient:	Frances M. Segarra Roman
Title:	EQB QA/QC Specialist Manager
Telephone:	787-767-8181, Ext. 3575

*Text has been revised as noted above.*



D. UFP-QAPP Worksheet # 6 ( 11) (Communication Pathways):

- a. Comment D-1: See Comments A-1, B-1, and C-1.
- b. Recommendation D-1: See Recommendations Comments A-1, B-1, and C-1.
- c. Recommendation D-2: The "QAPP Amendments" row should to be revised to include that all QAPP amendments will be also submitted to the QA/QC Specialist Manager for review and concurrence.

*Text has been revised as noted above.*

E. UFP- QAPP Worksheets # 20 (Field Quality Control Sample Summary Table):

- Comment E-1: The frequency of collection of the Field Duplicate and the MS/MSD are not clear.
- Recommendation E-1: For clarification purposes, the following frequencies are recommended so the table can be revised:

QA/QC Sample:	Frequency of Collection
Field Duplicate Sample	1 every 10 samples (10 %)
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	1 every 20 samples (20 %)

*The text has been revised to reflect a field duplicate frequency of 10%.*

**REVISED INTRINSIC BIODEGRADATION STUDY  
WORK PLAN – REVISION 1  
HEWLETT-PACKARD  
VOLUNTARY REMEDIAL ACTIONS  
SAN GERMAN, PUERTO RICO**

**PREPARED FOR:**

Hewlett-Packard Company  
Fort Collins, Colorado

**PREPARED BY:**

GZA GeoEnvironmental, Inc.  
Norwood, Massachusetts

April 2015  
Revised October 2015  
File No. 01.0024065.16

## TABLE OF CONTENTS

	<u>Page</u>
<b>1.0 INTRODUCTION.....</b>	<b>1</b>
<b>2.0 STATUS OF IB WORK PLAN IMPLEMENTATION .....</b>	<b>1</b>
2.1 IB WORK PLAN OBJECTIVES .....	1
2.1.1 Remedial Action Objectives .....	1
2.1.2 Monitoring Objectives .....	2
2.2 GROUNDWATER MONITORING AND SAMPLING.....	2
<b>3.0 DESCRIPTION OF PROPOSED PILOT TEST .....</b>	<b>3</b>
3.1 ERD PILOT TEST OBJECTIVE.....	3
3.2 ERD PILOT TEST OVERVIEW .....	3
3.3 INJECTION WELLS .....	4
3.4 PERMITS .....	4
3.4.1 DNER Permit .....	4
3.4.2 Injection Permit.....	4
3.5 SUBSTRATE COMPOSITION AND LOADING.....	4
3.6 BIOAUGMENTATION .....	5
<b>4.0 MONITORING .....</b>	<b>5</b>
4.1 MONITORING OF GROUNDWATER ELEVATIONS.....	5
4.2 PERFORMANCE MONITORING .....	5
4.3 SAMPLING PARAMETERS .....	5
4.4 SAMPLING FREQUENCY .....	6
4.5 DATA EVALUATION.....	6
<b>5.0 REPORTING .....</b>	<b>7</b>





## **TABLE OF CONTENTS (Continued)**

### **TABLES**

TABLE 1	INTRINSIC BIODEGRADATION SAMPLING PROGRAM
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### **FIGURES**

FIGURE 1	SITE LOCUS
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FIGURE 2	SITE PLAN
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FIGURE 3	PROPOSED INJECTION WELL LOCATIONS
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### **APPENDICES**

APPENDIX A	SEPTEMBER 27, 2010 INTRINSIC BIODEGRADATION STUDY WORK PLAN
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APPENDIX B	LIMITATIONS
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APPENDIX C	SAFETY DATA SHEETS ABC <sup>®</sup> and KB-1 <sup>®</sup>
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APPENDIX D	QUALITY ASSURANCE PROJECT PLAN (QAPP) REVISION 4
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## **1.0 INTRODUCTION**



An Intrinsic Biodegradation Study Work Plan (IB Work Plan) was submitted by GZA GeoEnvironmental, Inc. (GZA) on behalf of Hewlett-Packard Company (Hewlett-Packard) on September 27, 2010 (see Appendix A for the September 2010 IB Work Plan) in support of the Voluntary Soil and Groundwater Remediation project at the Former Digital Equipment Corporation facility in San German, Puerto Rico (the “Site” – see Figures 1 and 2). The Puerto Rico Environmental Quality Board (PREQB) approved the IB Work Plan on October 29, 2010. This is the first revision of the IB Work Plan. The findings, opinions, conclusions, and recommendations presented in this report are subject to the Limitations provided in Appendix B.

The objective of the study is to evaluate whether Intrinsic Biodegradation is a suitable remedial strategy for managing contaminant migration at this Site. The IB Work Plan summarizes the Site history and setting and documents the effects of remedial efforts conducted by Hewlett-Packard and its predecessors on a voluntary basis between 1995 and the present. It describes Site conditions prior to shutdown of the Groundwater Containment and Treatment System (GWCTS) on November 1, 2010, particularly as they pertain to the implementation of intrinsic biodegradation, and outlines a proposed monitoring program designed to evaluate the effectiveness of the remedial approach.

This first revision of the IB Work Plan was requested by PREQB in its November 22, 2013 letter approving the installation of additional monitoring wells. Revisions to the work plan include routine sampling of groundwater from the newly-installed monitoring wells, changes to monitoring frequency and parameters for certain wells based on reported groundwater concentrations of chlorinated organic compounds (cVOCs) since GWCTS system shutdown, and a change from quarterly groundwater elevation monitoring to semi-annual, in addition to other minor revisions.

Additionally, in an effort to evaluate the efficacy of Enhanced Reductive Dechlorination (ERD) as an additional means of cVOC destruction at the site, this work plan revision includes a description of a proposed ERD pilot test.

## **2.0 STATUS OF IB WORK PLAN IMPLEMENTATION**

The following sections describe additional work at the Site since implementation of the IB Work Plan and proposed monitoring to further evaluate the IB as a remedial strategy. Additional background and description of the Site can be found in the IB Work Plan submitted on May 14, 2010.

### **2.1 IB WORK PLAN OBJECTIVES**

#### **2.1.1 Remedial Action Objectives**

The objective of the IB Work Plan is to evaluate whether intrinsic biodegradation of the residual cVOCs in groundwater can continue to reduce dissolved concentrations while

posing no additional risk to human or environmental receptors. To this end, the GWCTS, including all extraction wells at the Site, was deactivated on November 1, 2010, and groundwater elevations and VOC concentrations were monitored.



### 2.1.2 Monitoring Objectives

The principal objectives of groundwater monitoring for the IB Work Plan are three-fold: (i) to evaluate whether dissolved concentrations at the Site continue to decline; (ii) to monitor the relative concentrations of trichloroethene (TCE) and its daughter compounds, as well as other known biologically sensitive parameters, in order to confirm biodegradation is occurring and characterize the respective degradation pathway; and (iii) to monitor conditions at perimeter wells to evaluate the potential for off-Site migration of impacted groundwater. These continue to be the monitoring objectives with respect to this first revision of the IB Work Plan.

## 2.2 GROUNDWATER MONITORING AND SAMPLING

Groundwater monitoring wells have been gauged quarterly for groundwater elevation and sampled semi-annually for chemical concentrations to evaluate the groundwater flow pathways and cVOC concentrations. With the exception of well BR-308, which is a 6-inch diameter bedrock well, the monitoring wells consist of two-inch diameter wells that have been installed in the fill/alluvium, saprolite, and bedrock units. Each monitoring well is covered by a road box to protect it from vehicle traffic. A lockable well plug is installed in each monitoring well casing beneath the road box.

Two additional monitoring wells were installed along the municipal road to the north of the Puerto Rico Electric Power Authority (PREPA) property between February 14 and 21, 2012. The wells were installed as sentinel wells to monitor off-Site groundwater. Monitoring well GZ-601L is installed in the saprolite unit, and GZ-601R is installed in the bedrock unit. The locations of the wells are depicted on Figure 2. The wells were installed under the Department of Natural and Environmental Resources (DNER) permit O-FA-PRE11-SJ-00280-23032011 dated April 25, 2011 and with written permission from the municipality of San German dated December 14, 2011.

Four additional monitoring wells were installed between April 7 and 17, 2014. Due to difficulties drilling through an unexpectedly thick alluvium layer, the installation of a fifth additional monitoring well was delayed and completed on August 17, 2014. The monitoring wells installed in 2014 include a well couplet to the west of GZ-513R along state highway PR-360 (GZ-702U and GZ-702R, respectively), a well couplet (saprolite and bedrock) to the north of GZ-507R in the municipal road (GZ-701L and GZ-701R, respectively), and a bedrock monitoring well in the vicinity of closed well W-6 (GZ-703R) as shown on Figure 2. The wells were installed under the DNER permit O-FA-PRE11-SJ-00430-16122013 dated December 16, 2013 and with written permission from the municipality of San German dated November 20, 2013; the owner of the Site (PRIDCO) dated February 5, 2014; and the Puerto Rico Roads and Transportation Authority dated January 28, 2014.

In addition, between November 1 and November 12, 2013, extraction wells W-1, W-7 and W-8 were decommissioned in accordance with the DNER permit O-FA-PSP07-SJ-00099-

28052013 issued on October 8, 2013. PREQB issued a letter of concurrence with the decommissioning on March 20, 2013.



Groundwater levels have been measured quarterly at up to 54 monitoring wells using a water level indicator, except for the groundwater elevations at 6 locations (GZ-507R, GZ-508R, GZ-509R, GZ-510R, GZ-512R, and GZ-513R) that are measured using pressure transducers. The monitoring wells at these locations have been closed; however, pressure transducers were installed prior to the well abandonment to provide ongoing information about groundwater elevations. Groundwater elevations at the Site have remained relatively consistent for the past three years.

Groundwater samples have been collected from approximately 30 to 54 monitoring wells (depending if it was a semi-annual or biennial sampling event) to monitor groundwater cVOC concentrations as well as IB parameters<sup>1</sup>. The groundwater samples were submitted to TestAmerica of Tallahassee, Florida and Savannah, Georgia and the results have been certified by a Puerto Rico-certified chemist and have been presented to PREQB in applicable semi-annual reports.

Groundwater gauging and sampling will continue to be performed to assess IB as a remedial strategy. Proposed gauging and sampling associated with the IB study are defined in Section 4.0 – Monitoring.

### **3.0 DESCRIPTION OF PROPOSED PILOT TEST**

#### **3.1 ERD PILOT TEST OBJECTIVE**

The objective of the proposed pilot test is to evaluate the efficacy of ERD as a means to improve cVOC destruction at the Site, with the additional goal of enhancing dissolution and degradation of residual source mass in the saprolite formation in the OW-307 area and the fill formation in the OW-101 area. This pilot test includes the installation of three dedicated injection wells and the injection of carbon-amended water and bioaugmentation culture into the new wells.

The proposed ERD pilot test is in addition to the Site-wide IB study, which will continue during and after the ERD pilot test. Proposed data collection will be of benefit in evaluating the effectiveness of both remedial strategies.

#### **3.2 ERD PILOT TEST OVERVIEW**

ERD would involve electron donor injection to enhance dissolved-phase cVOC destruction in areas that have relatively sustained TCE concentration. ERD pilot injections are proposed in the vicinity of saprolite monitoring well OW-307 and in the vicinity of well OW-101, which is screened in the fill deposit. Bioaugmentation, if deemed necessary, is proposed via addition of a concentrated culture of dechlorinating bacteria containing *Dehalococcoides*.

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<sup>1</sup> The IB parameters included dissolved iron, sulfate, methane, ethane, ethene, nitrate, total organic carbon (TOC), chloride, and the field parameters dissolved oxygen (DO), oxidation-reduction potential (ORP), and pH. Certain wells were analyzed for cVOCs, TOC, and the field parameters DO, ORP, and pH.

*Dehalococcoides* are a group of microorganisms known to provide complete de-chlorination of chlorinated ethenes to non-toxic ethene.

### 3.3 INJECTION WELLS

Three injection wells will be installed for the use of injecting carbon-amended water and bioaugmentation culture into the subsurface. Two shallow injection wells, IW-2 and IW-3, will be approximately 30 to 40 feet deep<sup>2</sup> and located 20 to 30<sup>3</sup> feet east and upgradient of monitoring well OW-307, parallel with estimated groundwater contours. One shallow injection well, IW-1, will be approximately 15 to 20 feet deep<sup>4</sup> and located 20 to 30 feet east and upgradient of monitoring well OW-101. The approximate locations of the injection wells are shown in Figure 3. Permits will be obtained for the installation of the proposed injection wells and for the proposed subsurface injections, as outlined below.

### 3.4 PERMITS

#### 3.4.1 DNER Permit

The injection wells for the ERD pilot test will be installed under the appropriate DNER permit. GZA is currently preparing the documentation to submit the permit application following the approval by PREQB of this revised IB Work Plan. Similar to the 2012 and 2014 well installations, we are requesting a concurrence letter from PREQB to assist in the DNER permit process.

#### 3.4.2 Injection Permit

The injections associated with the ERD pilot test will be conducted under the appropriate PREQB underground injection permit. GZA is currently preparing the documentation to submit the permit application following the approval of the IB Work Plan – Revision 1 by PREQB.

### 3.5 SUBSTRATE COMPOSITION AND LOADING

The electron donor carbon substrate we propose to inject is Anaerobic Biochem (ABC<sup>®</sup>, Redox Tech, LLC, Cary, North Carolina, USA – see Appendix C for safety data sheet). ABC contains C14 to C18 fatty acids derived from hydrolysis of vegetable oils. ABC is completely soluble in water, and includes ethyl lactate, a “green” co-solvent which also serves as a fast-reacting carbon substrate. Ethyl lactate helps dissolve the fatty acids and solubilize the cVOCs, increasing their bioavailability to degrading microbes. The substrate loading goal of the ERD pilot test is to inject as much carbon as practical into the saprolite and fill; therefore, we plan to inject at approximately four times the stoichiometric substrate demand<sup>5</sup>. Following installation of the injection wells, batches of water (currently estimated

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<sup>2</sup> Injection wells will be installed to depth of saprolite, and screened below the water table.

<sup>3</sup> Wells will be installed as far upgradient as practical, considering topography.

<sup>4</sup> Injection well will be installed to depth of fill and screened below the water table.

<sup>5</sup>As estimated using the Environmental Security Technology Certification Program (ESTCP) Substrate Design Tool, <https://www.serdp-estcp.org/Program-Areas/Environmental-Restoration/Contaminated-Groundwater/ER-200627/>

at 500 gallons per batch for a total of 2,000 gallons) amended with approximately 18% (by weight) ABC® will be injected into each well under low pressure, based on well acceptance<sup>6</sup>.

### 3.6 BIOAUGMENTATION



A Bio-Trap® Sampler (Microbial Insights, Inc., Knoxville, Tennessee, USA) was deployed into OW-307 in conjunction with the Fall 2014 sampling event at the Site. The sampler will be retrieved in conjunction with the Fall 2015 sampling event, and analyzed using molecular biological tools to estimate Site populations of dechlorinating microbes and quantities of their cVOC-degrading enzymes. If dechlorinating bacteria and enzymes are very low, GZA proposes bioaugmentation with specialized bacterial cultures (KB-1®, SiREM Labs, Guelph, Ontario, Canada – see Appendix C for safety data sheet) in conjunction with the next sampling event following the ABC injections at the Site.

## 4.0 **MONITORING**

### 4.1 MONITORING OF GROUNDWATER ELEVATIONS

Groundwater elevations across the Site under non-pumping conditions will continue to be monitored throughout the IB study and ERD pilot test to assess the direction of groundwater flow in each of the hydrogeologic units. A groundwater level indicator will continue to be used to measure the depth to groundwater in each of the on-Site and newly installed off-Site monitoring wells and when applicable from the proposed injection wells. Due to the relative consistency of groundwater level measurements since implementation of the IB Work Plan, groundwater level measurements will be performed semi-annually, reduced from the previous quarterly schedule.

### 4.2 PERFORMANCE MONITORING

Based on the constituents of concern at the Site, a key parameter to be measured during the IB study and ERD pilot test will be cVOC concentrations in groundwater monitoring wells across the Site. Groundwater samples from a subset of monitoring wells screened in the alluvium/fill, saprolite, and bedrock units at the Site will also be analyzed for the suite of biodegradation screening parameters described in Section 4.3 to evaluate the effectiveness of both the IB study and ERD pilot test<sup>7</sup>.

### 4.3 SAMPLING PARAMETERS

cVOC concentrations will be evaluated in groundwater monitoring wells across the Site. The following cVOCs will be analyzed: carbon tetrachloride, chlorobenzene, chloroethane,

---

<sup>6</sup> We anticipate being able to gravity inject at an average rate of approximately one gallon per minute. If conditions are encountered which indicate that the actual injection rate is significantly different than the estimated rate, the concentrations of ABC additive will be adjusted accordingly to maximize the quantity of additive injected and the volume of the treated aquifer. That is, a more dilute or more concentrated solution of ABC additive will be injected. The mass of ABC injected will be the same

<sup>7</sup> QAPP Worksheet #18: Sampling Locations and Methods describes the monitoring well subsets that will be analyzed for biodegradation screening parameters.





chloroform, chloromethane, dibromochloromethane, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, dichlorodifluoromethane, 1,1-dichloroethane, 1,2-dichloroethane, cis-1,2-dichloroethene, trans-1,2-dichloroethene, 1,1-dichloroethene, 1,2-dichloropropane, cis-1,3-dichloropropene, trans-1,3-dichloropropene, methylene chloride, 1,1,1,2-tetrachloroethane, 1,1,2,2-tetrachloroethane, tetrachloroethene, 1,1,1-trichloroethane, 1,1,2-trichloroethane, trichloroethene, trichlorofluoromethane, 1,2,3-trichloropropane, and vinyl chloride<sup>8</sup>. In addition, the groundwater from a subset of wells will be monitored for additional biodegradation indicator parameters. The additional parameters include dissolved iron, methane, ethane, ethene, and total organic carbon (TOC). The field parameters dissolved oxygen (DO), oxidation reduction potential (ORP), and pH will be measured at all wells.

#### 4.4 SAMPLING FREQUENCY

Groundwater samples from monitoring wells screened in the alluvium/fill, saprolite, and bedrock units at the Site will be analyzed for the suite of parameters described in the previous section to evaluate the effectiveness of the IB study and ERD pilot test.

Proposed changes to the sampling frequency and parameters are based on the concentrations of cVOCs exhibited by the groundwater collected since implementation of the IB Work Plan. As documented in Table 1, 31 of the wells, including the proposed injection wells, will be sampled on a semi-annual basis, and the remaining 26 wells will be sampled biennially (once every other year). Based on their location and the current trends in cVOC concentrations, 16 wells will be measured for additional biodegradation indicator parameters as documented in Table 1.

Further details regarding sample collection methods, sample preservation and handling, chain-of-custody procedures, analytical procedures, and field and laboratory quality assurance/quality control will be provided in the site-specific Quality Assurance Project Plan (QAPP) Revision 4 that is included as Appendix D. The QAPP, which documents the manner in which quality assurance and quality control activities will be implemented throughout the study, is composed of the following elements:

- Description of project tasks, data quality objectives, and management,
- Description of data acquisition and management,
- Description of assessments, responses, and oversight, and
- Description of data validation, verification, and usability.

#### 4.5 DATA EVALUATION

The data collected from the monitoring wells across the Site will be evaluated throughout the study period to continue to assess the effectiveness of IB. Additionally, the data will be used to evaluate the efficacy of ERD. The groundwater monitoring data will be reviewed to continue to evaluate groundwater flow patterns.

---

<sup>8</sup> Other VOCs will be analyzed and reported in accordance with standard analytical methodology.

## **5.0 REPORTING**



Status reports will be prepared on a semi-annual basis for submission to PREQB with a copy to United States Environmental Protection Agency - Region 2. As is currently being performed, each status report will cover a six-month period (January through June and July through December). The Semi-Annual Progress Reports will contain the following information:

- Description of the type and frequency of monitoring activities conducted;
- Summary of data obtained during the reporting period, including groundwater elevation data, analytical data reports, and tables and charts of relevant groundwater parameters;
- Status of response operations and description of significant modifications;
- Description of issues which may affect the performance of the IB study and ERD pilot test and corrective actions to be conducted; and
- Planned activities for next reporting period.

## **TABLES**

**TABLE 1**  
 Intrinsic Biodegradation Sampling Program  
 Hewlett-Packard Voluntary Remediation Project  
 San German, Puerto Rico

File No. 01.0024065.16

Page 1 of 2

10/14/2015

WELL ID	SAMPLING FREQUENCY		PARAMETERS	
	Semi-Annual	Biennial	VOCs	VOCs & IB Indicators
<b>Overburden (Wells screened in Fill/Alluvium)</b>				
GZ-501U		x	x	
GZ-503U		x	x	
GZ-504U	x		x	
GZ-506U		x	x	
GZ-511U		x	x	
GZ-515U		x		x
GZ-519U	x			x
GZ-702U <sup>4</sup>	x		x	
OW-101	x			x
OW-105		x	x	
OW-304U		x	x	
OW-305I		x	x	
OW-305U		x	x	
OW-402U	x		x	
OW-404U	x		x	
WB-1U	x		x	
WB-2U	x		x	
IW-1 <sup>5</sup>	x			x
<b>Saprolite</b>				
DEC-204O		x	x	
GZ-501L	x		x	
GZ-502L		x	x	
GZ-503L	x			x
GZ-504L	x			x
GZ-505L		x	x	
GZ-601L <sup>4</sup>		x	x	
GZ-701L <sup>4</sup>	x		x	
OW-1		x	x	
OW-101L	x		x	
OW-102		x	x	
OW-301		x		x
OW-304L	x			x
OW-307	x			x
OW-401		x	x	
OW-402L		x	x	
OW-403L		x	x	
OW-404L		x	x	
OW-405		x	x	
OW-407		x	x	
OW-408		x	x	
WB-1L	x		x	
WB-2L	x		x	
WB-3L	x			x
WB-4L	x			x
IW-2 <sup>5</sup>	x			x
IW-3 <sup>5</sup>	x			x

**TABLE 1**  
Intrinsic Biodegradation Sampling Program  
Hewlett-Packard Voluntary Remediation Project  
San German, Puerto Rico

File No. 01.0024065.16

Page 2 of 2

10/14/2015

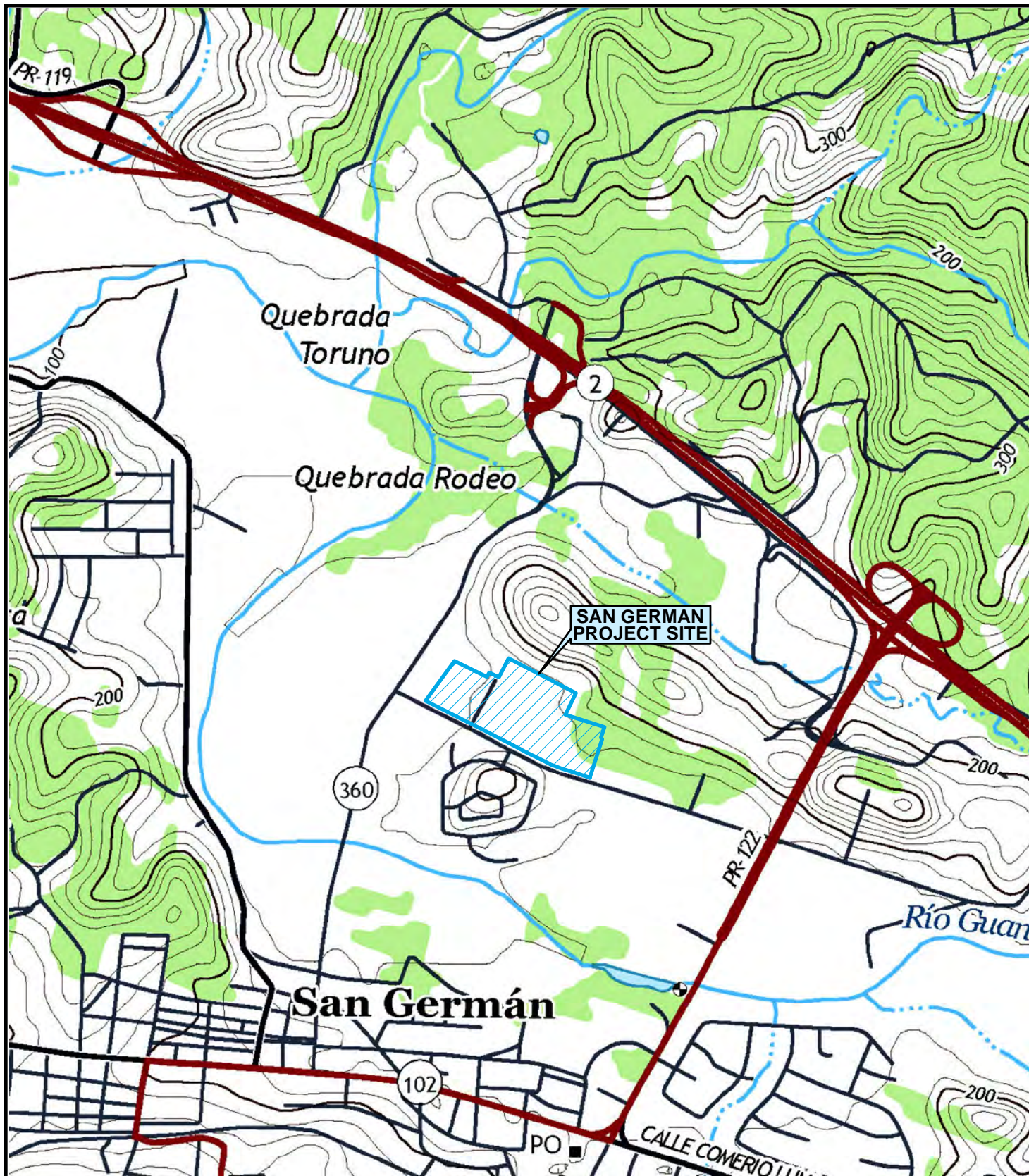
WELL ID	SAMPLING FREQUENCY		PARAMETERS	
	Semi-Annual	Biennial	VOCs	VOCs & IB Indicators
<b>Bedrock</b>				
BR-308		x	x	
DEC-203R		x	x	
GZ-504R	x			x
GZ-505R	x			x
GZ-506R	x			x
GZ-601R <sup>4</sup>	x		x	
GZ-701R <sup>4</sup>	x		x	
GZ-702R <sup>4</sup>	x		x	
GZ-703R <sup>4</sup>	x		x	
OW-304R	x		x	
OW-402R		x	x	
OW-404R	x		x	

Notes:

1. "VOCs" indicates analysis limited to only chlorinated volatile organic compounds (cVOCs), and the field parameters dissolved oxygen (DO), pH, and oxidation-reduction potential (ORP).
2. "VOCs & IB Indicators" set indicates analysis of cVOCs, dissolved iron, methane, ethene, ethane, TOC, and the field parameters DO, pH, and ORP.
3. Wells highlighted in gray represent a change in sampling frequency and/or parameters from the October 2010 Work Plan.
4. Denotes well installed subsequent to the October 2010 IB Work Plan submission and approval.
5. Denotes well to be installed and sampled as part of ERD pilot test.

## FIGURES





SOURCE : SCANNED USGS TOPOGRAPHIC QUADRANGLES  
DISTRIBUTED BY THE U.S. GEOLOGICAL SURVEY (USGS);  
SAN GERMAN, PUERTO RICO QUADRANGLE.



PROJ. MGR.: JAC  
DESIGNED BY: JY  
REVIEWED BY: CAL  
OPERATOR: PCF  
DATE: 01-28-2015

## SITE LOCUS PLAN

HEWLETT-PACKARD VOLUNTARY REMEDIATION PROJECT  
SAN GERMAN, PUERTO RICO

JOB NO.  
01.0024065.16  
FIGURE NO.  
1



© 2015 - GZA GeoEnvironmental, Inc. Z:\23,000-24,999\24065\24065-16 JV\Figures\GIS\MXD\24065-14\_SitePlan\_FIG2.mxd, 1/23/2015, 2:52:11 PM, patrick.linnerty



**LEGEND**

- ABANDONED BEDROCK EXTRACTION WELL
- MONITORING WELL SCREENED IN FILL
- MONITORING WELL SCREENED IN ALLUVIUM
- MONITORING WELL SCREENED IN SAPROLITE
- MONITORING WELL SCREENED IN BEDROCK
- ABANDONED MONITORING WELL
- SOIL VAPOR EXTRACTION WELL
- APPROXIMATE PROPERTY BOUNDARY
- ELEVATION CONTOUR (2012 USGS TOPOGRAPHIC QUADRANGLE)

**NOTES:**

1) THIS MAP CONTAINS THE ESRI ArcGIS ONLINE WORLD IMAGERY MAP SERVICE, PUBLISHED DECEMBER 12, 2009 BY ESRI ARCS SERVICES AND UPDATED OFTEN. THIS SERVICE USES UNIFORM NATIONALLY RECOGNIZED DATUM AND CARTOGRAPHY STANDARDS AND A VARIETY OF AVAILABLE SOURCES FROM SEVERAL DATA PROVIDERS.

2) THE CATCH BASINS, DRAIN LINES, DITCH AND VARIOUS LABELS WERE APPROXIMATELY LOCATED FROM THE FOLLOWING TWO PLANS:  
- PLAN PROVIDED BY PEDRO PANZARDI & ASSOCIATES ENTITLED "SITE PLAN WITH ELEVATIONS OF SEWER SYSTEM", DATED: 03-11-1988, ORIGINAL SCALE: 1:500 OR 1"=41', SHEET NO.: 3-OF-7, PROJECT NO.: 881, DRAWING NO.: 10-1-1.  
- PLAN PROVIDED BY DIGITAL EQUIPMENT CORPORATION (DEC) OF SAN GERMAN, PUERTO RICO ENTITLED "PLAN & PROFILE", DRAWN BY: VELAZQUEZ, DATED: 01-25-1987, ORIGINAL SCALE: 1:500 OR 1"=41'.

3) REFERENCE ELEVATIONS AND WELL LOCATIONS WERE SURVEYED BY JOSE O. QUINONES MORET, BS, PLS OF PUERTO RICO ON AUGUST 18, 2014. ELEVATIONS ARE RELATIVE TO LAJAS 2 (PID AB9841, NAVD 29) IN SABANA GRANDE, PUERTO RICO WITH ELEVATION 200.27 FEET. COORDINATES ARE PROVIDED IN NAD 83.

0 25 50 100 150

SCALE IN FEET

UNLESS SPECIFICALLY STATED BY WRITTEN AGREEMENT, THIS DRAWING IS THE SOLE PROPERTY OF GZA GEOENVIRONMENTAL, INC. (GZA). THE INFORMATION SHOWN ON THE DRAWING IS SOLELY FOR THE USE BY GZA'S CLIENT OR THE CLIENT'S DESIGNATED REPRESENTATIVE FOR THE SPECIFIC PROJECT AND LOCATION IDENTIFIED ON THE DRAWING. THE DRAWING SHALL NOT BE TRANSFERRED, REUSED, COPIED, OR ALTERED IN ANY MANNER FOR USE AT ANY OTHER LOCATION OR FOR ANY OTHER PURPOSE WITHOUT THE PRIOR WRITTEN CONSENT OF GZA. ANY TRANSFER, REUSE, OR MODIFICATION TO THE DRAWING BY THE CLIENT OR OTHERS, WITHOUT THE PRIOR WRITTEN EXPRESS CONSENT OF GZA, WILL BE AT THE USER'S SOLE RISK AND WITHOUT ANY RISK OR LIABILITY TO GZA.

INTRINSIC BIODEGRADATION WORK  
PLAN - REVISION 1  
SAN GERMAN, PUERTO RICO

SITE PLAN

PREPARED BY: <b>GZA GeoEnvironmental, Inc.</b> Engineers and Scientists www.gza.com		PREPARED FOR: <b>HEWLETT-PACKARD COMPANY</b>	
PROJ MGR: JY	REVIEWED BY: CAL	CHECKED BY: CAL	FIGURE
DESIGNED BY: JY	DRAWN BY: EMD	SCALE: 1 INCH = 50 FT	2
DATE: 01-23-2015	PROJECT NO. 01.0024065.16	REVISION NO.	

Source: Esri, DigitalGlobe, G







## **APPENDIX A**

SEPTEMBER 27, 2010 INTRINSIC BIODEGRADATION STUDY WORK PLAN

September 29, 2010  
File No. 01.0024065.10



Ms. Frances M. Segarra Roman  
Puerto Rico Environmental Quality Board  
Land Pollution Branch  
Cruz A. Matos Environmental Agencies Building  
1375 Ponce de Leon Ave.  
San Juan, Puerto Rico 00926-2604

Re: Intrinsic Biodegradation Study Work Plan  
Hewlett-Packard Company Voluntary Remediation Project  
San German, Puerto Rico

Dear Ms. Roman:

1 Edgewater Drive  
Norwood  
Massachusetts  
02062  
781-278-3700  
FAX 781-278-5701  
www.gza.com

GZA GeoEnvironmental, Inc. (GZA), on behalf of Hewlett-Packard Company, is pleased to submit to the Environmental Quality Board the enclosed response to comments and revision of the work plan for evaluating intrinsic biodegradation at the above referenced location. Appendix D of the work plan (Quality Assurance Project Plan (QAPP), Revision 3, May 2010) has not been edited, and, therefore, has not been resubmitted.

We, respectfully, request your review and approval of the work plan and QAPP. Please contact the undersigned or Mr. Paul Paschke (Hewlett-Packard Company) at 970-898-0573 or paul.paschke@hp.com, if you have any questions.

Very truly yours,

GZA GEOENVIRONMENTAL, INC.

A handwritten signature in blue ink, appearing to read "John A. Colbert".

John A. Colbert  
Senior Project Manager

A handwritten signature in black ink, appearing to read "I. Richard Schaffner, Jr.".

I. Richard Schaffner, Jr.  
Senior Technical Specialist

A handwritten signature in blue ink, appearing to read "Matthew J. Barvenik".

Matthew J. Barvenik  
Senior Principal

Attachments:

GZA's Responses to PREQB's Comments  
Intrinsic Biodegradation Study Work Plan (September 2010)

cc: File  
Aviles, Jesse; United States Environmental Protection Agency Region II  
Justiniano, Eng. Louis Rivera; Puerto Rico Industrial Development Company  
Paschke, Paul; Hewlett-Packard Company



**GZA's Response to PREQB's Comments**  
on  
Intrinsic Biodegradation Study Work Plan  
Voluntary Remediation Project  
Hewlett-Packard Company (HP)  
San German, Puerto Rico  
PRD-991291857  
prepared by  
GZA GeoEnvironmental, Inc., of  
Norwood, Massachusetts

Dated: September 27, 2010

**The following comments are from an Evaluation of the Intrinsic Biodegradation Study Work Plan from María V. Rodríguez Muñoz, Project Manager of the LPCA division of PREQB dated September 1, 2010.**

1. PAGE 6 – The method the work plan listed for VOC in the narrative was the 8260C. The correct id for this method should be 8260B since the one written on the work plan is not listed on the EPA web site containing the current SW-846 on-line methods. Until method 8260B is not revised, it continues to be the latest version.

*According to the EPA website, 8260C was an approved new method starting in August 2006. Please see the following website for more information: [http://www.epa.gov/osw/hazard/testmethods/sw846/new\\_meth.htm](http://www.epa.gov/osw/hazard/testmethods/sw846/new_meth.htm). We prefer to use this method as it is the method currently employed by the lab for the Site and the lab we use is phasing out 8260B. We are happy to discuss.*

2. PAGE 7 – Add at the end of the last paragraph of section 2.7, specifically after the word “reactivated”, the following: **“with the concurrence of the PREQB. A report will be submitted to the PREQB with \_\_\_\_\_ of the completion of the project.”**

*This has been changed. A report will be submitted within three months of the completion of the Intrinsic Biodegradation evaluation. This will most likely be included as part of or as an appendix to a Semi-Annual Status Report.*

3. Question: How HP will monitor if the plume began to migrate off site?

*This is described in sections 4.3 – Monitoring of Groundwater Elevations, 4.6.2 – Groundwater Elevations, and 4.7 – Contingency Plan. Section 4.7 describes the conditions that would lead to the implementation of the contingency plan and examples of the additional remedial response actions that may be considered. Due to the nature of the project, we do not believe that identification of a predetermined method and/or plan would be appropriate at*





*this time. We will be monitoring groundwater elevation and groundwater quality at numerous interior and perimeter wells, including:*

- *OW-301 (southeastern corner of the Site screened in the saprolite formation);*
- *GZ-515U (southwestern corner of the Site screened in the alluvium formation);*
- *WB-1U (western edge of Site screened in fill);*
- *GZ-504L (northwestern corner of the Site screened in the saprolite formation);*
- *GZ-504R (northwestern corner of the Site screened in bedrock); and*
- *OW-1 (northeastern corner of the Site screened in the saprolite formation).*

*(Note: At this time wells in the northeastern corner of the Site are often dry; however, under non-pumping conditions ample water may become present for sample collection.)*

*By monitoring groundwater elevations across the Site, we will be able to approximate groundwater flow direction. By monitoring groundwater quality across the Site, we will be able to assess if border wells experience a significant and sustained increase in concentrations of contaminants of concern.*

4. Question: For how long the GWTS will be shutdown or deactivated?

*The length of shutdown will be dependent on the findings of the evaluation. Because groundwater travels relatively slowly at the Site, it will take a considerable, but as yet unquantifiable amount of time to be able to evaluate whether or not intrinsic biodegradation is a suitable remedial option for managing VOC migration at the Site.*

5. Notice that the property has no guard. This was also observed by the contractors. HP should explain how you will work to ensure that the GWTS is not vandalized?

*A gate and padlock has been added to the Site. HP plans to have a contractor visit the Site on a monthly basis to inspect the property and functionally test and activate pumps and motors associated with the GWTS. The GWTS will be decommissioned to the point where it will be secure and could be reactivated with minimal effort.*

6. PAGE 11:

a. SECOND PARAGRAPH:

- i. When the narrative indicates that there were no private well in the “vicinity of the site”, what does this means? At what distance you are referring?



*A thorough survey of public and private wells that would be in hydrologic communication with the Site has previously been conducted. The RCRA Facility Investigation Summary Report submitted to EQB in July 1995 describes the public and private well survey conducted as part of Phase II investigations at the Site. Pertinent excerpts follow:*

*“Based on the findings of our past studies, we have not identified any likely on-going receptors of chlorinated ethene contamination from the site.”*

*“GZA identified data suggesting the presence of 23 public and private drinking water wells in the site area. Human exposure via public supplies is not considered likely for 21 of these wells, because the wells are located upstream of likely flow paths, and across the Guanajibo River from the site. As such these wells are believed to be hydrologically isolated from the site. The exposure pathway completion for the two remaining wells (located about 300 feet south of the site at the El Convento Housing District) was unknown, but thought unlikely, based on GZA’s understanding of groundwater flow directions from the site. The findings of GZA’s water well research were subsequently independently confirmed by a study conducted by V. S. Rodriguez and associates (VSR).”*

*With respect to the two remaining wells mentioned in the previous paragraph, “The presence of a privately owned water supply well at the El Convento Housing District was further supported by the recollections of a local well driller. This well, however, has not been confirmed by site reconnaissance observations or a review of official records, including Water Franchise Permits...Furthermore, based on our interpretation of groundwater flow conditions, if these wells exist, they are expected to be upgradient or sidegradient of likely groundwater flow paths from the site under site pumping conditions.”*

*In February 2010, the PRASA well list was reviewed and the absence of groundwater extraction wells permitted under PRASA and in communication with the Site was verified.*

*Additionally, On March 16, 2010, GZA confirmed with Francisco Aguilar at the neighboring Johnson and Johnson property that there is no pumping of groundwater at that facility. GZA is in the process of reviewing additional sources to update and verify the findings of the previous well surveys.*



- ii. Explain in the narrative in more detail what “additional responses” will be proposed in the case of migration off-site of the plume.

*We have added a reference to the contingency plan (section 4.7).*

b. THIRD PARAGRAPH:

- i. Instead of writing on the first sentence “The primary environmental receptors in the vicinity”, the sentence should read “**The primary surface water bodies in the vicinity**”. The word “**downstream**”, if applies (i.e. if it is only referring to the water bodies downstream from the site), should be added at the end of the first paragraph.

*We have made changes to the paragraph.*

- ii. The word *receptor* should be replaced by the word *target*.

*The word “target” has been used in this paragraph both in lieu of the word “receptor” and in addition to it.*

- iii. The last sentence of this paragraph is referring to human receptors. Basically, any concern with human receptors should be not only if migration off-site is occurring, but if there are private or public drinking water wells within a 4-mile radius from the site that may be impacted.

*See response to Question 6.a.i.*

7. PAGE 13:

a. SECTION 4.2:

- i. Replace the words “daughter compounds” by the words “degradation products or compounds”. Give examples of the “other known biologically active parameters”. Explain.

*“Daughter compounds” has been replaced by “degradation products” in the document. Other known biologically active parameters are total organic carbon, chloride, nitrate, dissolved oxygen, pH, oxidation-reduction potential, dissolved iron, sulfate, methane, ethane, and ethene. The reader has been referred to Section 4.4 that describes these biologically active parameters.*



8. PAGE 14:

a. SECOND PARAGRAPH:

- i. Explain the rationale of what is said on the last sentence of this paragraph for selecting a complete set or a subset of parameters. What is the subset of parameters versus the complete set of parameters?

*The rationale and the parameters included in the complete set and the limited set are given in Table 2. Table 3 is mistakenly referenced. This has been changed.*

9. When referring to a USEPA document in the narrative inside a parenthesis, include the ID number and the date of the publication.

*This has been changed.*

10. The work plan does not have a section on QA/QC samples, although it has a table mentioning them, that indicates how many and which ones will be collected during each sampling event. In particular it does not have an SOP on them indicating the rate of collection of them. This should be included. We suggest the following: one TB for VOC per shipment, one Field Blank per day of sampling (needs to be tested for all the parameters for which the rest of the samples will be collected). Equipment blanks are collected when using non-disposable sampling equipment and when decontamination of this equipment is performed in the field.

*The Quality Assurance Project Plan (QAPP) contained in Appendix D of the work plan contains the details of our QA/QC plans. The sampling SOP found in section Appendix D of the QAPP (3.1.2 – Sample Collection – Monitoring Wells, 3/2010, Rev. No. 5) describes the sampling procedure. Worksheet #20 of the QAPP gives the number of Trip Blanks (1 per cooler of VOA vials), field duplicates, and matrix spike/matrix spike duplicates. Worksheet #28 describes the frequency of QC samples (1/20 for field duplicates; 1/20 for MS/MSD).*

*We propose field QC samples be taken only for VOCs (one of the more sensitive analytes) because the other parameters are secondary lines of evidence, and, therefore, do not need additional field QC. Because the contaminants of concern at the Site are not pervasive in the environment, trip blanks would be enough to describe exposure that may occur. Forgoing field blanks also leads to a more conservative evaluation of Site conditions; therefore, our proposal is that no field blanks will be collected. Only trip blanks and matrix spike/matrix spike duplicates will be collected. Equipment blanks may be collected if sampling equipment is decontaminated and reused.*

**INTRINSIC BIODEGRADATION STUDY WORK  
PLAN  
HEWLETT-PACKARD  
VOLUNTARY REMEDIAL ACTIONS  
SAN GERMAN, PUERTO RICO**

**PREPARED FOR:**  
Hewlett-Packard Company  
Fort Collins, Colorado

**PREPARED BY:**  
GZA GeoEnvironmental, Inc.  
Norwood, Massachusetts

September 2010  
File No. 01.0024065.10

## TABLE OF CONTENTS

Page

	<b>1.0 INTRODUCTION</b>	<b>1</b>
	<b>2.0 BACKGROUND AND SITE DESCRIPTION</b>	<b>1</b>
	2.1 SITE LOCATION AND HISTORY	1
	2.2 GEOLOGIC AND HYDROGEOLOGIC SETTING	2
	2.3 SOURCE AREAS	3
	2.4 REMEDIAL GOALS	4
	2.5 REMEDIAL SYSTEMS	4
	2.6 GROUNDWATER MONITORING AND SAMPLING	6
	2.7 PERFORMANCE OF EXISTING REMEDIAL STRATEGY	6
	<b>3.0 EVALUATION OF INTRINSIC BIODEGRADATION AS A REMEDIAL APPROACH</b>	<b>7</b>
	3.1 EVIDENCE OF ONGOING BIODEGRADATION	8
	3.2 POTENTIAL RECEPTORS	9
	3.3 ALTERNATIVE REMEDIAL TECHNOLOGIES	11
	3.4 DISRUPTION TO SITE OPERATIONS	12
	3.5 APPLICABILITY OF INTRINSIC BIODEGRADATION	12
	<b>4.0 DESCRIPTION OF PROPOSED STUDY</b>	<b>12</b>
	4.1 REMEDIAL ACTION OBJECTIVES	12
	4.2 MONITORING OBJECTIVES	13
	4.3 MONITORING OF GROUNDWATER ELEVATIONS	13
	4.4 SAMPLING PARAMETERS	13
	4.5 SAMPLING FREQUENCY	14
	4.6 DATA EVALUATION	15
	4.6.1 Geochemical Evaluation	15
	4.6.2 Groundwater Elevations	15
	4.7 CONTINGENCY PLAN	16
	4.8 REPORTING	16
	<b>5.0 REFERENCES</b>	<b>17</b>

### TABLES

TABLE 1	INTRINSIC BIODEGRADATION BASELINE SAMPLING PROGRAM
TABLE 2	INTRINSIC BIODEGRADATION SAMPLING PROGRAM



## **TABLE OF CONTENTS (Continued)**

### **FIGURES**

FIGURE 1 SITE LOCUS

FIGURE 2 SITE PLAN

### **APPENDICES**

APPENDIX A LIMITATIONS

APPENDIX B VOC CONCENTRATION TREND ANALYSIS CHARTS

APPENDIX C VOC MOLARITY TREND ANALYSIS CHARTS

APPENDIX D QUALITY ASSURANCE PROJECT PLAN (QAPP)  
(SUBMITTED MAY 2010)



## **1.0 INTRODUCTION**



This Intrinsic Biodegradation Study Work Plan is submitted in support of the Hewlett-Packard Company (Hewlett-Packard) Voluntary Soil and Groundwater Remediation project at the Former Digital Equipment Corporation facility in San German, Puerto Rico (the “Site” – see Figures 1 and 2). The findings, opinions, conclusions, and recommendations presented in this report are subject to the Limitations provided in Appendix A.

The objective of the study is to evaluate whether Intrinsic Biodegradation would be a suitable remedial strategy for managing contaminant migration at this Site. The work plan summarizes the Site history and setting, and documents the effects of remedial efforts conducted by Hewlett-Packard and its predecessors on a voluntary basis between 1995 and the present. It describes current Site conditions, particularly as they pertain to the implementation of intrinsic biodegradation, and outlines a proposed monitoring program designed to evaluate the effectiveness of this remedial approach. Details regarding monitoring parameters, frequency of monitoring, data presentation, and reporting requirements have also been included in the work plan.

## **2.0 BACKGROUND AND SITE DESCRIPTION**

### **2.1 SITE LOCATION AND HISTORY**

The Site consists of approximately 18 acres, located on State Highway 362 in San German, Puerto Rico. It is located approximately 1,200 feet east of the Guanajibo River in a tributary drainage basin, and is bounded by a steep northeast to southeast trending ridge to the north and a smaller hill to the south. The topography generally slopes downward from the central portion of the Site towards the parking areas to the west and southeast with about 20 to 30 feet of relief.

The property is bounded to the north by undeveloped land, to the northwest and west by the Puerto Rico Electric and Power Authority (PREPA), to the south by State Road 362, and to the east by an industrial facility. The Site is owned by the Puerto Rico Industrial Development Company (PRIDCO), which leased the land to Digital Equipment Corporation (Digital) from July 1968 to 1992. Digital operated a printed wire board (PWB) and module assembly manufacturing facility at the Site, and in the mid-1970s, used trichloroethylene (TCE) in their Wave Solder Process as a degreaser and cleaning agent. Digital stopped using TCE in 1978, and terminated manufacturing operations at the Site in 1991. The facility was inactive until January 1993, when the Site was occupied by Circo Caribe. Circo Caribe manufactured PWBs at the Site until March 2001. In October 2001, PCB Horizon Technology Inc. leased the facility and began low-volume production of PWBs in November 2002. PCB Horizon Technology vacated the facility in 2005 and the facility remains vacant.





In preparation for the termination of Digital's lease of the facility, Digital completed two environmental investigations in 1992 and 1993. These investigations identified the presence of chlorinated ethenes (the presumed parent compound TCE and the assumed degradation product *cis*-1,2-dichloroethylene (1,2-DCE)) in the groundwater at the Site. The investigations concluded that TCE was likely to have been released from floor piping and wastewater trenches located at the facility production area. In response to these findings, Digital voluntarily implemented a remediation program.

Compaq purchased Digital in 1998, and assumed responsibility for the voluntary remediation efforts initiated by Digital. Subsequently, Hewlett-Packard merged with Compaq in 2002 and assumed responsibility for the operation of the remedial system.

## 2.2 GEOLOGIC AND HYDROGEOLOGIC SETTING

The Site geology generally consists of fill material overlying natural residual soils, which in turn overlie bedrock. The Site and vicinity were apparently filled and graded in the past. Areas likely to have been cut include the vicinity of topographic highs west of Building 1 and south of Building 5, whereas areas with the greatest fill include the western and central portions of the Site. The fill material ranges from stiff-to-hard clay and silt with up to 35 percent each sand and gravel, to medium-to-dense sand with up to 35 percent silt and clay and up to 35 percent gravel. The fill unit is absent in the south-central area of the Site and missing over most of the northern portion of the PREPA property. It is present at thicknesses of up to 23 feet across the central portions of the Site and the adjacent PREPA property, consistent with the presence of a generally east-west trending pre-development valley feature across the Site.

The natural residual soils consist of silt and clay soils underlain by saprolite formed by natural chemical weathering of the bedrock. The silt and clay layer appears discontinuous across the Site. The saprolite is typically denser than the clay and silt, has a greater gravel content, and has more evidence of primary rock textures and structures such as joints. It generally increases in density with depth, and has been differentiated from the bedrock by auger refusal.

The bedrock consists of altered mafic igneous rock, and was encountered at depths ranging from approximately 15 feet to greater than 50 feet below ground surface (bgs). The greatest depth of confirmed bedrock exists along the axis of the pre-development topographic valley in the central, northwest portion of the Site. Relatively shallow bedrock was observed in a soil boring near the southwest corner of Building 1, in the general vicinity of the pre-development topographic high.

Two groundwater systems are present at the Site; one appears to be perched within the shallow fill material with a water table ranging in depth from approximately 5 to 10 feet bgs, and the other is located in the saprolite and bedrock with a potentiometric surface ranging in depth from approximately 20 to 40 feet bgs. The depth of fill/alluvium ranges from approximately 10 to 30 feet bgs. Saprolite is located below the fill/alluvium layer, and its thickness ranges from approximately 10 to 40 feet. Bedrock is located below the



saprolite layer at approximately 20 to 60 feet bgs. A more detailed evaluation of the Site's hydrogeologic characteristics was presented in GZA's March 2003 Hydrogeologic Investigation Report, which assessed conditions across the Site with particular emphasis on the hydraulic connections between the fill, saprolite, and bedrock units.

The report indicated that, in the western part of the Site, a shallow perched water bearing zone in the western portion of the parking lot is located in fill and alluvial soils overlying the saprolite layer. The alluvium material appears to act as an aquitard, limiting hydraulic connection between upper and lower layers. Phase I of the hydrogeologic investigation concluded that neither the fill nor the saprolite had been found to act as a significant migration pathway, and noted that under normal non-pumping conditions, flow of groundwater in the fill unit would be toward the west, with WB-1L representing downgradient conditions at the Site. The flow pattern has a semi-radial component affected by zones of more pervious backfill in the parking lot and infiltration of surface water from storm drains and the backfill material.

Phase II of the hydrogeologic investigation studied bedrock conditions in the central "A Street" area of the Site, and noted that the bedrock aquifer in the region between extraction wells W-1 and W-6 has extensive and widespread fracturing in at least the upper 40 feet of bedrock. The saprolite layer was observed to be extensively hydraulically connected to the bedrock aquifer in this region. It was further noted, based on observations during a rainfall event, that there was direct recharge to the fill above the saprolite, but this recharge was not observable in the saprolite or bedrock.

Phase III of the hydrogeologic investigation determined that the elevation of the bedrock appears to rise slightly from the center of the western parking lot to the western property boundary, and that the bedrock on the western property boundary is fractured but to a lesser extent than in the central part of the Site. Subsequently, Phase IV of the investigation, which included a 2-week period of non-pumping conditions at the Site, concluded that under non-pumping conditions, groundwater in the saprolite/bedrock unit would generally flow toward the pre-development valley in the center of the Site and then northwestward toward the Guanajibo River.

### 2.3 SOURCE AREAS

The historic TCE concentration data indicate two primary and one secondary general source areas. The highest concentration source area appears to be in the saprolite unit in the general area south of the Plant Chemical Storage Area (potentially also including TCE releases from former wastewater trenches under the production facility). The second primary source area, in the overburden fill unit, appears to be located just south of the Existing Hazardous Waste Storage Area, and may reflect previous use as a drum storage area. The secondary (and lower concentration) source area is represented by groundwater impacts in the fill and saprolite units in the vicinity of a stormwater catch basin near the western property line, and appears to reflect historical seepage of unrelated release events through the bottom of the western boundary catch basin.



## 2.4 REMEDIAL GOALS

As stated in GZA's July 1995 RCRA Facility Investigation (RFI) Summary Report, the objectives of the voluntary remediation program at the Site were to:

- Contain and treat volatile organic compound (VOC)-containing groundwater; and
- Remediate VOC concentrations in the vadose zone soils to reduce impact to the groundwater.

To meet these goals, a Soil Vapor Extraction (SVE) system and a Groundwater Containment and Treatment System were installed at the Site. Details about the operation of these systems are provided in the following section.

## 2.5 REMEDIAL SYSTEMS

A soil vapor extraction (SVE) system was operated at the Site between October 1995 and November 2004. The system consisted of three SVE wells that extracted VOC vapors from the soil vadose zone in the front loading dock area of the Site, which was the only confirmed source area in soils for subsurface VOC contamination identified during investigations in the early 1990s. The SVE system was operated for over eight years, until three years of data indicated that the concentrations of VOCs in extracted vapor samples had achieved asymptotic levels. Based on these data, Hewlett-Packard received EQB's concurrence to deactivate and decommission the SVE system in a letter dated October 25, 2004. The SVE system was deactivated on November 11, 2004 and decommissioning was completed in February 2005.

Since 1995<sup>1</sup>, VOC plume migration at the Site has been managed by a Groundwater Containment and Treatment System (GWCTS) currently consisting of the following components:

- Groundwater extraction wells (W-1, W-7, and W-8);
- Extraction well piping network;
- Groundwater treatment system (GWTS);
- Groundwater monitoring wells;
- A telemetry system; and
- An alarm auto-dialer.

There are three wells currently incorporated into the groundwater extraction system: W-1, W-7 and W-8. The original function of these wells was to provide process

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<sup>1</sup> It is noted that prior to 1995, groundwater extraction was also generally being conducted by the on-site water production wells.



water for facility operations when the facility was occupied and in production. However, there are currently no manufacturing operations at the Site, so there is no demand for process water. If water is needed in the future for operations, municipal water can be provided. Therefore, the extraction wells are no longer essential for facility operations.

From a remediation standpoint, the original function of these wells was to create and maintain a capture zone and deliver the extracted water to the GWTS for VOC removal. These wells are no longer required under the remediation plan presented below.

From a historic background perspective, the primary extraction wells used to create the capture zone during the recent past have been W-8 and W-7, because they had a greater influence on the downgradient groundwater capture zone than extraction well W-1. Of these two primary extraction wells, W-8 was the lead well given its proximity to the higher VOC-concentration areas at the Site. This well is located in the western parking lot, east of the basketball court, and is 350 feet deep. Extraction well W-7 is located in the southwestern corner of the western parking lot, toward which groundwater flowed prior to the installation of extraction well W-8. It was generally operated to extract impacted groundwater in this area, which is outside the capture zone of extraction well W-8. The total open depth of extraction well W-7 is 161 feet, based on field observations during the installation of a new pump and motor in the well during the week of June 8, 2009<sup>2</sup>.

Extraction well W-1 has historically been a backup well that was typically operated when: (1) a primary extraction well (W-7 or W-8) was not operational, or (2) the primary extraction wells could not provide sufficient water for facility process water use. Extraction well W-1 is 350-feet deep and is located in the northern area of the Site, in a shed north of Building 2 in the Plant Chemical Storage Area. It has currently replaced W-7 as one of the primary extraction wells at the Site.

The operation of the groundwater extraction wells is controlled by water level sensors in a 5,000-gallon equalization tank that is part of the GWTS. The GWTS is located in the northeastern portion of the Site. Groundwater treatment at the GWTS consists of filtration by basket strainers and sand filters for the removal of particulates and precipitated iron followed by carbon adsorption for the removal of VOCs, in particular TCE and 1,2-DCE which are the main contaminants of concern. Treated groundwater is preferentially routed to the facility for discharge directly to the sanitary sewer under Hewlett-Packard's Puerto Rico Aqueduct and Sewer Authority (PRASA) Authorization Discharge Authorization (AUA-E-06-313-018) if the facility does not require all the groundwater extracted<sup>3</sup>.

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<sup>2</sup> It is believed that the original depth of extraction well W-7 was on the order of 350 feet, but this total depth has never been confirmed. The current, shallower open depth of 161 feet is believed to be the result of a borehole collapse below that point.

<sup>3</sup> Manufacturing operations are not currently being conducted at the facility. Therefore, all of the treated groundwater is being discharged directly to the PRASA sewer.



A telemetry system monitors the flow of groundwater from each extraction well and the flow of treated water from the GWTS. The GWTS auto-dialer alarm calls a local subcontractor if a GWTS system alarm is activated.

Influent and effluent samples are collected monthly and are tested for VOCs, including TCE and 1,2-DCE to evaluate the potential for breakthrough of the activated carbon, which is determined by increasing VOC concentrations in the effluent sample. Table 1 presents the results of influent and effluent sampling conducted during the last six months of 2009 in comparison to the results collected during the first two quarters of system operation. The liquid-phase carbon is replaced before breakthrough is reached. The spent activated carbon is shipped off-Site by a licensed waste management company for proper disposal or recycling.

## 2.6 GROUNDWATER MONITORING AND SAMPLING

Groundwater monitoring wells are gauged quarterly for groundwater elevation and sampled semi-annually for chemical concentrations to assess the groundwater capture zone of the GWTS. With the exception of well BR-308, which is a 6-inch diameter bedrock well, the monitoring wells consist of two-inch diameter wells that have been installed in the fill/alluvium, saprolite, and bedrock units. Each monitoring well is covered by a road box to protect it from vehicle traffic. A lockable well plug is installed in each monitoring well casing beneath the road box.

The groundwater level is measured quarterly at approximately 50 monitoring wells using a water level indicator, except for the groundwater elevations at six locations (GZ-507R, GZ-508R, GZ-509R, GZ-510R, GZ-512R, and GZ-513R) that are measured using pressure transducers. The monitoring wells at these locations have been closed; however, pressure transducers were installed prior to the well abandonment to provide ongoing information about groundwater elevations.

Groundwater samples are collected from approximately 25 monitoring wells on a semi-annual basis to monitor groundwater VOC concentrations, and the samples are submitted for laboratory analysis by US Environmental Protection Agency (USEPA) Method 8260C. The analytical results are certified by a Puerto Rico-certified chemist and are presented to PREQB in semi-annual reports.

## 2.7 PERFORMANCE OF EXISTING REMEDIAL STRATEGY

The primary objective of the existing groundwater extraction and treatment system was to control potential off-site migration of the plume, and also to reduce the dissolved VOC concentrations to the extent practical. Evaluation of Site conditions indicates that TCE concentrations greater than 100 µg/L continue to be detected only in the immediate vicinity of the source areas (in wells OW-304L/304R near the plant chemical storage area and in wells OW-305U and to a lesser extent GZ-502L at the hazardous waste storage



area<sup>[1]</sup>), and along A Street in the center of the Site (OW-101), suggesting that impacted groundwater has not significantly migrated away from these source areas. While one explanation for this limited migration is the impact of hydraulic control, it is also possibly due to naturally occurring processes that are biodegrading the chlorinated VOCs (cVOCs) in the subsurface and thus limiting the size of the dissolved plume.

The groundwater extraction and treatment system has been operated on a voluntary basis for 14 years. Over this period, influent concentrations to the system have remained relatively consistent and have averaged 0.025 mg/L TCE, resulting in removal of less than one liter of TCE per year.

Review of the current Site conditions indicates the present remedial approach is not a cost-effective or efficient means of removing VOCs from the subsurface. In addition, the effectiveness of natural biodegradation in controlling impacted groundwater cannot be evaluated while the extraction wells are in operation. In fact, operation of the groundwater extraction system may be limiting the effectiveness of intrinsic biodegradation, which is an anaerobic pathway at this Site, because the increased mass flux of oxygenated water through the respective formation has the effect of inhibiting reductive dechlorination.

As documented in the following sections of this work plan, there is evidence that intrinsic biodegradation of the cVOCs is occurring within the subsurface and may be successful at reducing cVOC concentrations in the groundwater. Therefore, GZA proposes to deactivate the groundwater extraction and treatment system during the period of time necessary to collect data and evaluate the effectiveness of intrinsic biodegradation as a remedial approach at this Site. At the conclusion of this evaluation, GZA will make a recommendation on whether the groundwater extraction and treatment system should be reactivated with the concurrence of the PREQB. A report will be submitted to the PREQB within three months of the completion of the Intrinsic Biodegradation evaluation. This will most likely be included as part of or as an appendix to a Semi-Annual Status Report.

### **3.0 EVALUATION OF INTRINSIC BIODEGRADATION AS A REMEDIAL APPROACH**

An intrinsic biodegradation remedial approach refers to reliance on naturally occurring biological processes (within the context of a controlled and monitored site cleanup approach) to achieve site specific remedial objectives within a timeframe that is reasonable, compared to that of other remedial strategies. As defined by USEPA, the process depends on indigenous microflora to degrade contaminants without any remedial amendments (EPA, 2006, EPA/625/R-06/015). This approach is used *in situ* and takes

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<sup>[1]</sup> TCE concentrations greater than 100 µg/L are also detected on occasion in WB-1L, near the western perimeter of the Site.



advantage of naturally-occurring processes to degrade cVOCs, with careful monitoring to demonstrate the ongoing processes are protective of potential receptors.



Based on GZA's experience at several similar projects over the last decade, intrinsic biodegradation can be considered as an effective remedial approach for cVOCs in ground water when one or more of the following conditions are present at the Site.

- Intrinsic biodegradation is observed or strongly expected to be occurring.
- Potential receptors, if present, in the vicinity of the contamination will not be adversely impacted.
- VOCs are present that cannot be easily and cost-effectively removed and will require a long-term remedial effort.
- Alternative remedial technologies are not cost effective or are technically impractical.
- Alternative remedial technologies pose added risk by transferring or spreading contamination.
- Minimal disruption of facility operations or infrastructure is desired.

The following sections provide information regarding conditions at the San German Site with respect to the criteria listed above.

### 3.1 EVIDENCE OF ONGOING BIODEGRADATION

Biodegradation of chlorinated solvents occurs under anaerobic groundwater environments in the presence of microbial species capable of degrading these compounds through respiration to various degradation compounds. Biologically reductive dehalogenation (BRD) typically results in the sequential breakdown of the chlorinated parent compound TCE to its degradation compound 1,2-DCE, which in turn is dechlorinated to yield vinyl chloride, which in turn is dechlorinated to yield ethene. The rate of biodegradation is controlled by several factors related to the availability of required elements (e.g., an organic carbon "food" source), nutrients, and growth factors necessary for the viability of the microbial population.

Intrinsic biodegradation can be evaluated using a "line of evidence" approach, including the following:

- Primary Line of Evidence - Documentation of loss of contaminants through reviewing historical trends in contaminant concentration and distribution in conjunction with Site geology and hydrogeology, to show that reduction in total mass of contaminants is occurring.
- Secondary Line of Evidence - Evaluation of the change in concentration and distribution of geochemical and biological indicator parameters that have been correlated to biodegradation pathways.

At this Site, evaluation of the groundwater TCE concentrations over time indicates a generally decreasing temporal trend (primary line of evidence), particularly at the source areas of the Site, where concentrations are currently less than half of historical high



concentrations. Appendix B presents graphs of the historical concentrations of TCE, 1,2-DCE, and vinyl chloride (VC) over time for eighteen monitoring wells that have been routinely sampled. In general, the concentrations of TCE at the wells have exhibited a downward temporal trend. For example, TCE concentrations at well cluster OW-304, located near a primary source area, have decreased from greater than 70,000 µg/L to 6,300 µg/L in well OW-304L (screened in saprolite) and from greater than 15,000 µg/L to 1,000 µg/L in well OW-304R (screened in bedrock).

In addition, the data confirm increasing concentrations of 1,2-DCE relative to TCE, indicating that the TCE released by former operations is being degraded to its degradation by-product 1,2-DCE and suggesting that intrinsic biodegradation may be a viable option for remediation of the residual groundwater contamination. Given that one mole of TCE yields one mole of 1,2-DCE via a reductive dechlorination pathway, mass per volume measurements of each parameter are biased by the mass difference between both chemical compounds (*i.e.*, while one mole of TCE yields one mole of 1,2-DCE, one gram of TCE yields less than one gram of 1,2-DCE) due to the replacement of the heavier chlorine atom with a lighter hydrogen atom. To normalize the data for the purpose of evaluating the TCE to 1,2-DCE transformation path, GZA converted these compounds from mass per volume (concentrations) to their molar equivalencies. Molarity trend analyses (Appendix C) more clearly illustrate a shift toward 1,2-DCE dominance at the Site. In the absence of a release of both TCE and 1,2-DCE, the relationship between TCE and 1,2-DCE is that of parent to degradation compound, where the degradation compound represents a dechlorination product of the parent compound. At this Site, data from most wells show a generally higher concentration of 1,2-DCE than of TCE and some (GZ-504R, GZ-502L, and OW-404L) with a recent trend demonstrating an increasing temporal dominance of 1,2-DCE over TCE. Four wells (OW-101, GZ-506R, OW-304R, and OW-304L) show a higher TCE concentration over 1,2-DCE. However, of the four wells, three show the TCE concentration approaching the 1,2-DCE concentration. The one exception, GZ-506R, is located between a historic source area and the extraction wells; thus the recent change in the primary extraction wells could explain why this particular well exhibits an increase in TCE.

Given the above observations it can be concluded that the most likely explanation for the 1,2-DCE dominance at the Site is that biodegradation is occurring via a reductive dechlorination pathway, converting TCE to 1,2-DCE (because there has been no known release of 1,2-DCE on Site).

### 3.2 POTENTIAL RECEPTORS

In evaluating intrinsic biodegradation, one of the primary considerations is the potential effects on potential receptors at and around the release area. At this Site, there are no current human receptors on the property, which has been unoccupied since 2005. Access to casual human visits (*i.e.*, trespassers) is limited by a fence around the property and security measures provided by PRIDCO. Extended access by construction or utility

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<sup>4</sup> *i.e.*, the lowest TCE concentration detected at this well for more than a decade.





workers, while possible, is not anticipated in the next several years. Changes in Site use are not anticipated during the period of evaluation of the feasibility of implementing a biodegradation remedy. If such changes do occur during the evaluation, an assessment of the potential risks to such receptors will be performed at that time based on the nature of the changed use. Currently, the nearest potential human receptors in the vicinity of the Site include workers at the facilities located east and west of the Site, and residents located to the south of the Site. Since the cVOCs at this Site are present in groundwater at depths of greater than 5 feet below grade, direct contact is not considered a complete exposure pathway. Instead, the risk to potential receptors would be inhalation risks associated with vapor intrusion into occupied buildings. In order to evaluate these risks, the maximum dissolved groundwater concentrations in the perimeter wells at the Site were compared to numerical values for TCE (5 µg/L) and cis-1,2-DCE (210 µg/L) listed in Table 2b of EPA's November 2002 OSWER *Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils* (EPA530-D-02-004). Per the guidance document, these numerical values represent "conservative 'generic' attenuation factors that reflect generally reasonable worst-case conditions for a first-pass screening of groundwater [...]."

Along the western perimeter of the Site, the screening evaluation was based primarily on data from wells GZ-504U, WB-1U, OW-404U, WB-2U, WB-3L, WB-4L, and GZ-515U, which represent groundwater conditions in the uppermost aquifer at each of these locations (i.e. in the case of the GZ-504 cluster, data from GZ-504U was used to represent the uppermost fill aquifer; in the case of WB-3L, the data was used even though the well is screened in saprolite because it is the most shallow groundwater data available for this location). The comparison indicates that with the exception of WB-2U, where the maximum TCE concentration of 8 µg/L slightly exceeds the screening value of 5 µg/L, the dissolved concentrations in the wells listed above were below the screening values for TCE and cis-1,2-DCE. It should be noted that the 8 µg/L concentration reported for WB-2U may be anomalous, since the well was sampled on 14 occasions, and TCE concentrations on the 13 other occasions were consistently below the detection limit of 1 µg/L. Therefore, based on this screening evaluation, the potential inhalation risk to receptors west of the Site does not represent an unacceptable risk.

Along the eastern perimeter of the Site, the maximum dissolved TCE and cis-1,2-DCE concentrations from perimeter wells OW-301, OW-407, and OW-1 and W-5 were found to be below the USEPA screening values, indicating that the potential inhalation risk to receptors east of the Site does not represent an unacceptable risk.

Data from the southern boundary of the Site are more limited, and less conclusive. Historic maximum TCE concentrations along the south-eastern portion of the southern boundary, represented by monitoring wells OW-302 and OW-303A, which are screened in highly weathered rock, exceed the screening value of 5 µg/L. However, there were only two data points for OW-302, and review of the OW-303A data indicate that the average TCE concentration over 19 sampling episodes was 4 µg/L, below the screening value of 5 µg/L. Based on these data, inhalation risks associated with groundwater in the south-eastern part of the Site does not represent an unacceptable risk.



Shallow groundwater concentrations along the southern boundary west of “A” Street are represented by monitoring wells OW-405, GZ-516U, and GZ-515U. Of these, the TCE concentration in well OW-405 was 60 µg/L in 2003, but insufficient water was present in the well to confirm this result during 8 subsequent sampling events; TCE and cis-1,2-DCE concentrations in the latter two wells have been consistently below the EPA screening values. Additionally, it is anticipated that under non-pumping conditions groundwater flow will be towards the center of the site and northwestward toward the Guanajibo River. Based on the data from GZ-516U and GZ-515U the potential inhalation risk represented by groundwater concentrations along the southern boundary of the Site does not represent an unacceptable risk. As part of the proposed biodegradation monitoring program additional data is scheduled to be collected from monitoring wells GZ-515U and OW-405 and the potential for risk associated with vapor intrusion will be further evaluated.

GZA’s 1995 risk characterization evaluated the risks associated with ingestion and dermal contact with groundwater from private water supply wells. It concluded that there were no registered private wells in the vicinity of the Site. In January 2010, GZA conducted a review of PRASA’s records and concluded that there remain to be no registered private water supply wells used for drinking water in the vicinity of the Site. Groundwater elevations and perimeter well concentrations along the southern and western Site boundaries will be closely monitored during the intrinsic biodegradation study as described later in this document, and changes of groundwater flow direction or increases in perimeter concentrations will be evaluated to assess their potential impact on nearby receptors. If data indicate that groundwater is migrating off-site and may have an impact on potential off-site receptors, GZA will propose additional responses as described in Section 4.7 (Contingency Plan). Also, the potential for off-site impacts will be taken into consideration when evaluating whether intrinsic biodegradation can be successfully implemented as a long-term remedial approach.

The primary surface water body in the vicinity of the Site is the Guanajibo River, located downgradient and approximately 1/3 mile west and ¼ mile south of the property. As in the case of human receptors or targets, data from the perimeter monitoring wells will be evaluated during the intrinsic biodegradation study, and data suggesting increased off-Site migration will be incorporated into the decision process regarding the suitability of intrinsic biodegradation as a remedial alternative.

### 3.3 ALTERNATIVE REMEDIAL TECHNOLOGIES

Remedial technologies, including soil vapor extraction to address vadose zone contamination and groundwater pumping and treatment to address groundwater VOC concentrations, have been implemented at the Site. The groundwater extraction and treatment system has been operational for over 14 years, and the data demonstrate that the residual source material is not being effectively removed using this technology.



Therefore, we are recommending that the alternative technology of intrinsic bioremediation be evaluated. It should be noted that this work plan also includes a contingency to consider other alternative technologies, such as in-situ chemical oxidation or enhanced bioremediation, if the intrinsic biodegradation study indicates the need for additional remediation.

### 3.4 DISRUPTION TO SITE OPERATIONS

Disruption to Site operations is not a consideration at this Site because the facility is no longer in operation.

### 3.5 APPLICABILITY OF INTRINSIC BIODEGRADATION

The Site meets three of the key conditions for implementation of intrinsic biodegradation: 1) there is evidence that ongoing bioremediation is occurring at the Site; 2) use of biodegradation is unlikely to pose a significantly increased risk to receptors; and 3) there are still contaminants in the groundwater after many years of treatment. It is GZA's opinion and the data support that mass removal of VOCs via the GWCTS is de minimis. Therefore, the more cost efficient remedial option of intrinsic bioremediation, which is expected to be at least as effective, should be tried. In fact, it is not only clear that intrinsic bioremediation is currently removing VOC mass from the Site, but it is likely to become more effective once the current operation of the groundwater extraction system is deactivated. This is because intrinsic biodegradation is an anaerobic pathway on Site and the increased mass flux of oxygenated water through the formation due to the pumping has the effect of inhibiting reductive dechlorination; as such, intrinsic biodegradation should be enhanced relative to its current level of effectiveness if the groundwater extraction system is deactivated. Therefore, GZA proposes to deactivate the existing system during the implementation of the intrinsic biodegradation study as described in the next section of this report.

## **4.0 DESCRIPTION OF PROPOSED STUDY**

### 4.1 REMEDIAL ACTION OBJECTIVES

The objective of the proposed study is to evaluate whether, in the absence of any additional remedial technologies, intrinsic biodegradation of the residual cVOCs in groundwater can continue to reduce dissolved concentrations while posing no additional risk to human or environmental receptors. To this end, the study proposes to deactivate the GWTS including all extraction wells at the Site and monitor groundwater elevations and VOC concentrations as described below. It should be noted that the GWTS will remain at the site and be ready for reactivation if the need arises and data collected indicate that reactivation is warranted.



## 4.2 MONITORING OBJECTIVES

The principal objectives of groundwater monitoring are three-fold: (i) to evaluate whether dissolved concentrations at the Site continue to decline; (ii) to monitor the relative concentrations of TCE and its degradation compounds, as well as other known biologically sensitive parameters described in Section 4.4, in order to confirm biodegradation is occurring and the respective degradation pathway; and (iii) to monitor conditions at perimeter wells to evaluate the potential for off-Site migration of impacted groundwater.

## 4.3 MONITORING OF GROUNDWATER ELEVATIONS

Groundwater elevations across the Site under non-pumping conditions will be monitored throughout the study to assess the direction of groundwater flow in each of the hydrogeologic units. A groundwater level indicator will be used to measure the depth to groundwater in each of the on-Site monitoring and extraction wells following shutdown of the extraction wells. Groundwater levels from the adjacent property north of the Site will be collected via the pressure transducers installed at locations GZ-507R, GZ-508R, GZ-509R, GZ-510R, GZ-512R, and GZ-513R. Groundwater level measurements will be performed weekly for the first month, monthly for six months, and quarterly thereafter. The frequency of gauging may vary depending on groundwater rebound response and other factors.

## 4.4 SAMPLING PARAMETERS

Based on the constituents of concern at the Site, a key parameter to be measured during the study will be cVOC concentrations in groundwater monitoring wells across the Site. These data will be used to evaluate whether concentrations in each of the geologic units continue to decrease over time, and to monitor any potential off-Site migration.

Other chemical indicators representative of ongoing biodegradation will also be measured during the study. The process of biodegradation for cVOCs is largely based upon microbial respiration, during which cVOCs serve as electron acceptors to receive the electrons released during the metabolism of organic carbon, the electron donors. During this process (dehalorespiration), microbes gain energy from the consumption (oxidation) of electron donors coupled to the utilization (reduction) of electron acceptors. Dissolved oxygen (DO), which can serve as a terminal electron acceptor (TEA) and limit cVOC dechlorination by competing with the cVOCs for hydrogen and volatile fatty acids (VFAs)<sup>5</sup>, will be measured during each sampling event. In general, DO measurements of less than 0.5 ppm suggest that anaerobic conditions conducive for cVOC dechlorination may exist. Under anaerobic conditions (*i.e.*, in the absence of DO), electron acceptors

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<sup>5</sup> Note that hydrogen and VFAs are the “food” that drive reductive dechlorination. Without these electron donors, dechlorination cannot proceed via a dehalorespiration pathway.



such as nitrate, ferric iron, and sulfate are respectively utilized for nitrate reduction, ferrogenic, and sulfate reduction. Each of these pathways competes with cVOCs for hydrogen and VFAs, so additional groundwater samples will be collected for analysis of nitrate, ferrous (iron II), and sulfate. Other parameters that will be measured include the total organic carbon (TOC) concentration, which is a primary parameter that drives cVOC dechlorination by providing electron donor to native soil bacteria; the oxygen-reduction potential (ORP)<sup>6</sup>, which serves as an indicator of the Redox conditions that control cVOC dechlorination; pH, which acts as a general indicator of conditions conducive for natural biota (dechlorinating microorganisms can be particularly sensitive to low pH conditions); and methane, which is an indicator of anaerobic, chemically reducing conditions that can support cVOC dechlorination.

Samples will also be analyzed for chloride, which is a dechlorination product of cVOCs, and for ethanes and ethenes, the end products of cVOC reductive dechlorination, whose presence would demonstrate that the reaction is proceeding to completion.

#### 4.5 SAMPLING FREQUENCY

Groundwater samples from monitoring wells screened in the alluvium/fill, saprolite, and bedrock units at the Site will be analyzed for the suite of geochemical parameters described in the previous section to evaluate the effectiveness of the intrinsic biodegradation process.

Prior to initiation of the intrinsic biodegradation study, the 47 monitoring wells listed on Table 1 will be gauged and sampled to establish baseline conditions at the Site. Subsequently, as documented on Table 2, 30 of the wells will be sampled on a semi-annual basis, and the remaining 17 wells (designated in bold font on Table 2) will be sampled biennially (once every other year), unless TCE concentrations in proximal wells begin to increase, in which case they would be sampled semi-annually<sup>7</sup>. Based on their location and the current trends in cVOC concentrations, wells will be measured for either a limited sub-set of parameters (35 wells) or for a complete set of parameters (12 wells) as documented on Table 2. Table 2 provides the rationale for the selection of semi-annual versus biennial sampling and limited versus complete parameters for each well. It also describes the difference between the limited set of parameters and the complete set.

Further details regarding sample collection methods, sample preservation and handling, chain-of-custody procedures, analytical procedures, and field and laboratory quality assurance/quality control will be provided in a site-specific Quality Assurance Project Plan (QAPP) that is included as Appendix D of this work plan. The QAPP, which

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<sup>6</sup> Theoretically, aerobic degradative activity occurs at a highly positive redox potential, while anaerobic microbial processes such as methanogenesis and sulfate reduction will occur at strongly negative redox potentials; however interpretation of redox potential field data in terms of microbial activity can be extremely challenging, as these measurements are due to complex interactions between chemical species present in the groundwater and microbial byproducts.

<sup>7</sup> Additionally, monitoring wells that are sampled semi-annually that indicate an increase in cVOC concentration may be sampled at intervals more frequently than semi-annually.



documents the manner in which quality assurance and quality control activities will be implemented throughout the study, is composed of the following elements:

- Description of project tasks, data quality objectives, and management,
- Description of data acquisition and management,
- Description of assessments, responses, and oversight, and
- Description of data validation, verification, and usability.

#### 4.6 DATA EVALUATION

The data collected from the monitoring wells across the Site will be evaluated throughout the study period to assess the effectiveness of intrinsic biodegradation for remediating cVOC in groundwater at the Site. In addition, the groundwater monitoring data will be closely reviewed to evaluate groundwater flow patterns in the absence of on-Site pumping.

##### 4.6.1 Geochemical Evaluation

The primary indicator of ongoing biodegradation at the Site is anticipated to be a decrease in dissolved TCE concentrations, with a concurrent increase in 1,2-DCE and other degradation products relative to TCE concentrations in groundwater. Therefore, the dissolved TCE, 1,2-DCE, and VC concentrations will be plotted with historic data to evaluate whether the general temporal decrease seen in the current data continues over time. The data will also be used to evaluate whether the 1,2-DCE dominance of the Site continues, indicating continued breakdown of TCE into its degradation products.

If the results of the primary lines of evidence are not conclusive, then additional geochemical data will be evaluated to assess whether secondary lines of evidence support ongoing intrinsic biodegradation. For example, decreasing ORP, DO, nitrate, ferric iron or sulfate values, or increasing ethane, ethene or chloride concentrations, would generally be indicative of conditions suitable for cVOC reductive dechlorination at the Site.

##### 4.6.2 Groundwater Elevations

Groundwater level monitoring data collected from the wells in each geologic zone will be evaluated to assess the change in flow patterns at the Site following shutdown of the groundwater treatment system. Hydrographs will be created to chart groundwater elevations until hydraulic conditions stabilize, following which the groundwater elevation data will be contoured on a semi-annual basis to assess flow direction in the fill, saprolite, and bedrock zones at the Site. Rebound of groundwater levels to natural conditions is anticipated to be slow; however, if the data suggest that flow patterns are drastically different from those anticipated, the frequency of groundwater level monitoring will be increased in order to provide a more complete understanding of groundwater flow



direction with respect to source areas and potential receptors. Additional measures to address changes in flow patterns are discussed in the contingency section below.



#### 4.7 CONTINGENCY PLAN

The objective of the contingency plan is to identify certain conditions that would likely warrant action based on the ongoing review of the data being collected. The groundwater levels and static contaminant concentrations may take time to reach equilibrium. Additionally, it may take several rounds of data collection after that time to gain clarity and a reasonable level of certainty that equilibrium was established, and as a result the establishment of temporal and/or spatial concentration trends will be a protracted process. However, if an unfavorable condition is observed and confirmed, corrective actions will be implemented. Unfavorable conditions that would be of particular concern include confirmed trends demonstrating significantly increasing concentration of TCE in groundwater migrating off-site, reversal of degradation product dominant concentrations, absence of supporting data regarding intrinsic biodegradation activity from secondary lines of evidence and/or evidence of plume advancement. If such conditions are suspected and confirmed, additional remedial response actions will be considered, including enhanced biodegradation or *in-situ* chemical oxidation for localized areas, activation of select groundwater extraction wells associated with the GWCTS, and/or other remedial approaches that are deemed appropriate for the given condition. Similarly, if groundwater contouring indicates that flow patterns are significantly different than anticipated, additional monitoring wells may be installed downgradient of Site in the direction of groundwater flow.

A section of each status report will be dedicated to an evaluation of areas of concern and/or potential conditions (trends) being observed along with an explanation of actions or potential actions to be implemented.

#### 4.8 REPORTING

Status reports will be prepared on a semi-annual basis for submission to PREQB with a copy to USEPA Region 2. As is currently being performed, each status report will cover a six-month period (January through June and July through December). The Semi-Annual Progress Reports will contain the following information:

- Description of the type and frequency of monitoring activities conducted;
- Summary of data obtained during the reporting period, including groundwater elevation data, analytical data reports, and tables and charts of relevant groundwater parameters;
- Status of response operations and description of significant modifications;
- Description of issues which may affect the performance of the remedial strategy and corrective actions to be conducted; and
- Planned activities for next reporting period.



## 5.0 REFERENCES

GZA GeoEnvironmental, Inc., December 1994. Draft Voluntary Interim Measure, Soil and Groundwater Remediation, Former Digital Equipment Corporation Facility, San German, Puerto Rico, Digital Project Number 05203018.

GZA GeoEnvironmental, Inc., July 1995. RCRA Facility Investigation (RFI) Summary Report, Former Digital Equipment Corporation Facility, San German, Puerto Rico, Digital Project Number 052-03018.

GZA GeoEnvironmental, Inc., August 1995. Final Draft, Remedial Systems Design Report for the Interim Measures Soil and Groundwater Remediation at the Former Digital Facility, San German, Puerto Rico.

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United States Environmental Protection Agency (EPA), November 2002. EPA530-D-02-004 Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils.

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## **TABLES**

WELL ID	SAMPLING PARAMETERS		COMMENTS
	IB Indicators	CVOCs	
Overburden (Wells screened in Fill/Alluvium)			
OW-304U	x	x	Last sampled March 2003 (9 events)
GZ-506U	x	x	Last sampled March 2004 (15 events)
OW-105	x	x	Last sampled June 2003 (14 events)
OW-402U	x	x	Last sampled December 2001 (5 events)
GZ-519U	x	x	Last sampled June 2003 (12 events)
GZ-504U	x	x	Last sampled March 2004 (1 event)
WB-1U	x		Last sampled March 2009 (19 events)
GZ-501U	x	x	Last sampled June 2003 (13 events)
GZ-511U	x	x	Last sampled June 2003 (12 events)
OW-404U	x		Last sampled September 2009 (42 events)
OW-305U	x	x	Last sampled March 2004 (15 events)
OW-305I	x		Last sampled September 2009 (27 events)
WB-2U	x	x	Last sampled March 2004 (14 events)
GZ-503U	x	x	Last sampled March 2002 (8 events)
OW-101	x		Last sampled September 2009 (38 events)
GZ-515U	x		Last sampled September 2009 (23 events)
Saprolite			
OW-301	x		Last sampled September 2009 (21 events)
OW-407	x	x	Last sampled June 2003 (9 events)
OW-408	x	x	Last sampled March 2007 (4 events)
DEC-204O	x	x	Last sampled February 1993 (2 events)
OW-1	x	x	Last sampled February 1993 (1 event)
OW-304L	x		Last sampled September 2009 (46 events)
OW-403L	x	x	Last sampled June 2003 (30 events)
OW-102	x	x	Last sampled June 2003 (18 events)
OW-402L	x	x	Last sampled June 2003 (4 events)
OW-101L	x	x	Never sampled
OW-307	x	x	Last sampled June 2002 (12 events)
OW-405	x	x	Last sampled February 1993 (1 event)
GZ-505L	x		Last sampled September 2009 (24 events)
GZ-503L	x	x	Last sampled June 2003 (15 events)
OW-401	x	x	Last sampled June 2003 (28 events)
GZ-502L	x		Last sampled September 2009 (26 events)
GZ-501L	x	x	Last sampled June 2003 (14 events)
GZ-504L	x	x	Last sampled June 2003 (12 events)
WB-1L	x		Last sampled September 2009 (30 events)
OW-404L	x		Last sampled September 2009 (44 events)
WB-2L	x		Last sampled September 2009 (26 events)
WB-3L	x	x	Last sampled June 2003 (14 events)
WB-4L	x		Last sampled September 2009 (25 events)
Bedrock			
DEC-203R	x	x	Last sampled June 2003 (8 events)
OW-304R	x		Last sampled September 2009 (27 events)
GZ-506R	x		Last sampled September 2009 (27 events)
OW-402R	x	x	Last sampled June 2003 (9 events)
BR-308	x	x	Last sampled September 2004 (32 events)
GZ-505R	x		Last sampled September 2009 (24 events)
OW-404R	x		Last sampled September 2009 (26 events)
GZ-504R	x		Last sampled September 2009 (24 events)

- Notes:
- “IB” indicates Intrinsic Biodegradation, and “CVOCs” indicates chlorinated volatile organic compounds.
  - “IB Indicators” include dissolved iron, sulfate, methane, ethane, ethene, TOC, chloride, and the field parameters DO, nitrate, ORP, and pH.
  - Well locations in *italics* are often dry, inaccessible, or otherwise not able to be sampled.  
BR-308 requires a generator in order for it to be sampled.

**TABLE 2**  
Intrinsic Biodegradation Sampling Program  
Hewlett-Packard Voluntary Remediation Project  
San German, Puerto Rico

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Page 1 of 2

WELL ID	CURRENTLY MONITORED	PROPOSED FREQUENCY		PARAMETERS		RATIONALE
		Semi-Annual	Biennial	Limited Set	Complete Set	
Overburden (Wells screened in Fill/Alluvium)						
OW-304U		x		x		Apparent decreasing [TCE] temporal trend
GZ-506U		x		x		[TCE] near ND
OW-105		x		x		Apparent decreasing [TCE] temporal trend and near ND
OW-402U		x			x	Apparent decreasing or stable [TCE] temporal trend
GZ-519U		x			x	Apparent stable [TCE] temporal trend
GZ-504U	Yes	x		x		Well apparently never sampled, well along property line
WB-1U	Yes	x		x		Apparent decreasing [TCE] temporal trend, well along property line
GZ-501U		x		x		Apparent decreasing [TCE] temporal trend
GZ-511U			x	x		Apparent decreasing [TCE] temporal trend
OW-404U	Yes		x	x		[TCE] near ND
OW-305U			x	x		Variable [TCE]
OW-305I	Yes	x		x		Apparent decreasing [TCE] temporal trend
WB-2U			x	x		ND for TCE
GZ-503U		x		x		[TCE] near ND
OW-101	Yes	x			x	Apparent decreasing or stable [TCE] temporal trend
GZ-515U	Yes		x	x		[TCE] near ND, well along property line
Saprolite						
OW-301	Yes		x	x		ND for TCE
OW-407			x	x		ND for TCE
OW-408	Yes		x	x		Well not always accessible
DEC-2040		x		x		Last sampled 1993 with TCE detected at 22 ppb
OW-1	Yes	x		x		Well not always accessible
OW-304L	Yes	x			x	Apparent stable or decreasing [TCE] temporal trend; monitoring well in source area along fracture line
OW-403L		x			x	Apparent stable [TCE] temporal trend; monitoring well along fracture line
OW-102			x	x		ND for TCE; monitoring well along fracture line
OW-402L		x		x		Limited Set – Apparent decreasing [TCE] temporal trend and monitoring well along fracture line
OW-101L		x			x	Monitoring well apparently never sampled; location along fracture line
OW-307		x			x	Apparent stable [TCE] temporal trend; monitoring well along fracture line
OW-405	Yes		x	x		Well not always accessible
GZ-505L	Yes	x		x		Apparent decreasing [TCE] temporal trend approaching ND
GZ-503L		x			x	Apparent stable [TCE] temporal trend
OW-401			x	x		Apparent decreasing [TCE] temporal trend
GZ-502L	Yes	x			x	Variable to decreasing [TCE] temporal trend
GZ-501L		x		x		Apparent decreasing [TCE] temporal trend approaching ND
GZ-504L			x	x		Near ND for TCE
WB-1L	Yes	x		x		Apparent decreasing [TCE] temporal trend
OW-404L	Yes		x	x		Apparent decreasing [TCE] temporal trend
WB-2L	Yes	x		x		Near ND for TCE
WB-3L			x	x		ND for TCE
WB-4L	Yes		x	x		Near ND for TCE

**TABLE 2**  
**Intrinsic Biodegradation Sampling Program**  
**Hewlett-Packard Voluntary Remediation Project**  
**San German, Puerto Rico**

WELL ID	CURRENTLY MONITORED	PROPOSED FREQUENCY		PARAMETERS		RATIONALE
		Semi-Annual	Biennial	Limited Set	Complete Set	
Bedrock						
DEC-203R		x		x		Apparent decreasing [TCE] temporal trend
OW-304R	Yes	x		x		Apparent decreasing [TCE] temporal trend; monitoring well in source area along fracture line
GZ-506R	Yes	x			x	Apparent increasing [TCE] temporal trend; monitoring well along fracture line
OW-402R		x			x	Apparent stable [TCE] temporal trend; monitoring well along fracture line
BR-308	Yes		x	x		Well not always accessible & apparent decreasing [TCE] temporal trend
GZ-505R	Yes		x	x		ND for TCE six consecutive rounds
OW-404R	Yes	x			x	Apparent stable or decreasing [TCE] temporal trend
GZ-504R	Yes	x		x		Low to ND for [TCE]

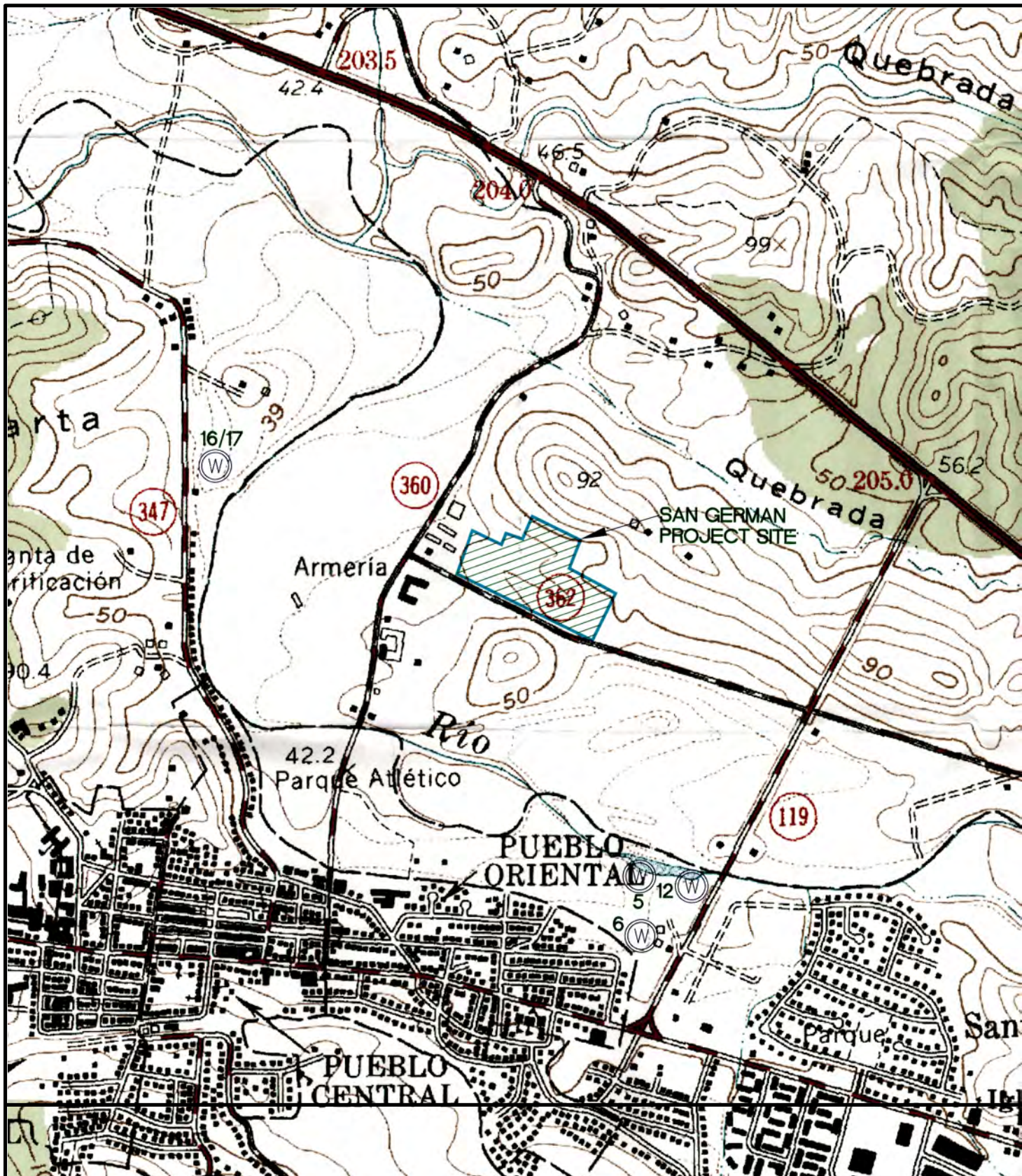
Notes:

- Monitoring program assumes an initial sampling round of all monitoring wells referenced herein to establish baseline conditions, followed by semi-annual sampling. Following baseline sampling, well locations in boldface will be sampled once every other year, unless [TCE]s in proximal wells become asymptotic above Maximum Contaminant Levels or begin to increase, in which case they would be sampled semi-annually.
- "IB" indicates intrinsic biodegradation; "TCE" indicates trichloroethene; "ND" indicates non detect above analytical reporting limit relative to constituent listed.
- "Limited Set" indicates analysis limited to only chlorinated volatile organic compounds (cVOCs), total organic carbon (TOC), and the field parameters dissolved oxygen (DO), pH, nitrate, and oxidation-reduction potential (ORP)
- "Complete Set" indicates analysis of cVOCs, dissolved iron, sulfate, methane, ethene, ethane, TOC, chloride, and the field parameters DO, pH, nitrate, and ORP.
- Well locations in *italics* are often dry, inaccessible, or otherwise not able to be sampled.
- For well locations being sampled for the "Limited Set" of parameters, if future monitoring results indicate a spike in TCE concentrations for two consecutive rounds, future analysis (same sampling frequency) will include the "Complete Set" of parameters.

## **FIGURES**



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SOURCE : SCANNED USGS TOPOGRAPHIC QUADRANGLES  
SUPPLIED BY U.S. GEOLOGICAL SURVEY;  
SAN GERMAN, PUERTO RICO QUADRANGLE.



PROJ. MGR.: JAC  
DESIGNED BY: JAC  
REVIEWED BY: JAC  
OPERATOR: GAS  
DATE: 05-05-2010

## SITE LOCUS PLAN

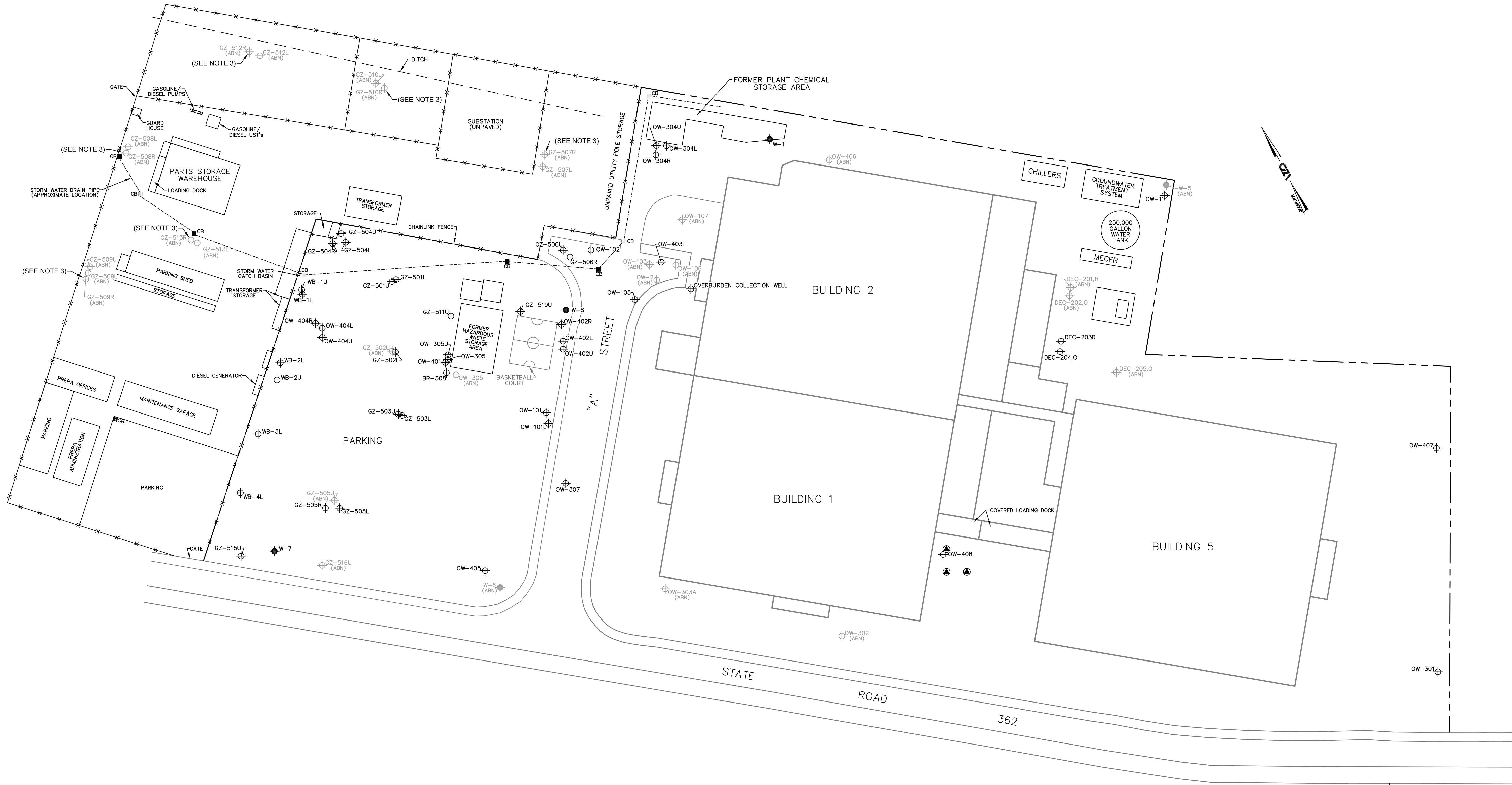
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INTRINSIC BIODEGRADATION WORK PLAN  
SAN GERMAN, PUERTO RICO

FIGURE NO.  
1



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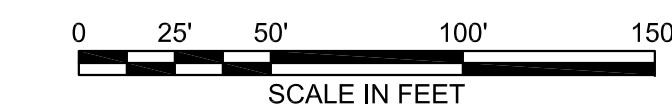


NOTES:

1. BASE MAP WAS DEVELOPED FROM THE FOLLOWING TWO PLANS:
  - (A) PLAN PROVIDED BY PEDRO PANZARDI & ASSOCIATES ENTITLED "SITE PLAN WITH ELEVATIONS OF SEWER SYSTEM," SHEET NO. 3 OF 7, PROJECT NO. B811, DWG. NO. 10-1.1, DATED 3/11/88, ORIGINAL SCALE: 1:500 OR 1" = 41'.
  - (B) PLAN PROVIDED BY DIGITAL EQUIPMENT CORPORATION (DEC), SAN GERMAN, PUERTO RICO, ENTITLED "PLAN & PROFILE" DRAWN BY YELAZQUES, DATED 1/23/87, ORIGINAL SCALE: 1:500 OR 1" = 41'.
2. THE LOCATIONS OF TEST BORINGS/WELLS WERE DETERMINED BY TAPE MEASURES AND "LINE-OF-SIGHT" OBSERVATIONS FROM EXISTING SITE FEATURES. THESE DATA SHOULD BE CONSIDERED ACCURATE ONLY TO THE DEGREE IMPLIED BY THE METHOD USED.
3. THE GROUNDWATER ELEVATIONS AT WELLS GZ-507R, GZ-508R, GZ-509R, GZ-510R, GZ-512R AND GZ-513R ARE MEASURED QUARTERLY USING PRESSURE TRANSDUCERS.

LEGEND

- W-8 BEDROCK EXTRACTION WELL
- OW-305U MONITORING WELL
- "U" INDICATES MONITORING WELL SCREENED IN FILL/ALLUVIUM
- "L" INDICATES MONITORING WELL SCREENED IN SAPROLITE
- "R" INDICATES MONITORING WELL SCREENED IN BEDROCK
- (ABN) INDICATES MONITORING WELL HAS BEEN ABANDONED
- SOIL VAPOR EXTRACTION WELL



INTRINSIC BIODEGRADATION STUDY WORK PLAN				
HEWLETT-PACKARD VOLUNTARY REMEDIATION PROJECT SAN GERMAN, PUERTO RICO				
SITE PLAN				
PREPARED BY: <b>GZA GeoEnvironmental, Inc.</b> Engineers and Scientists ONE EGGWATER DRIVE NORWOOD, MASSACHUSETTS 02062 (PH) 781-228-3700		PREPARED FOR:		
PRJ MGR: JAC	REVIEWED BY: MJB	CHECKED BY: JAC	FIGURE	
DESIGNED BY: ATD / JY	DRAWN BY: JPN / EMD	SCALE: 1" = 50 FEET	<b>2</b>	
DATE: 05-07-2010	PROJECT NO. 04.0024065.10	REVISION NO.		

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## **APPENDIX A**

### **LIMITATIONS**



## **LIMITATIONS**

1. The reported findings submitted in this report are based in part upon previous and recent data obtained from a limited number of samples from widely spaced subsurface explorations and monitoring wells. The nature and extent of variations between these explorations may not become evident until further investigation is performed. If variations or other latent conditions then appear evident, it will be necessary to re-evaluate the conclusions of this Report.
2. Water level readings have been made in the observation wells periodically and under conditions stated in the text. These data have been reviewed and interpretations have been made in the text of this Report. However, it must be noted that fluctuations in the level of the groundwater may occur due to variations in rainfall and other factors different from those prevailing at the time measurements were made.
3. Quantitative laboratory testing was performed as part of the site investigation and remediation work. Where such analyses have been conducted by an outside laboratory, GZA GeoEnvironmental, Inc. (GZA) has relied upon the data provided, and has not conducted an independent evaluation of the reliability of these data.
4. The findings contained in this Report are based in part upon various types of chemical data and are contingent upon their validity. These data have been reviewed and interpretations made in the Report. Some of these data were preliminary "screening" level data, and may have not been confirmed with quantitative analyses. Moreover, it should be noted that variations in the types and concentrations of contaminants and variations in their flow paths may occur due to seasonal water table fluctuations, past disposal practices, the passage of time, and other factors. Should additional chemical data become available in the future, these data should be reviewed by GZA, and the findings presented herein modified accordingly.
5. Chemical analyses have been performed for specific parameters during the course of this study, as detailed in the text. It must be noted that additional constituents not searched for during the current study may be present in soil and groundwater at the site.

## **APPENDIX B**

### **VOC TREND ANALYSIS CHARTS**

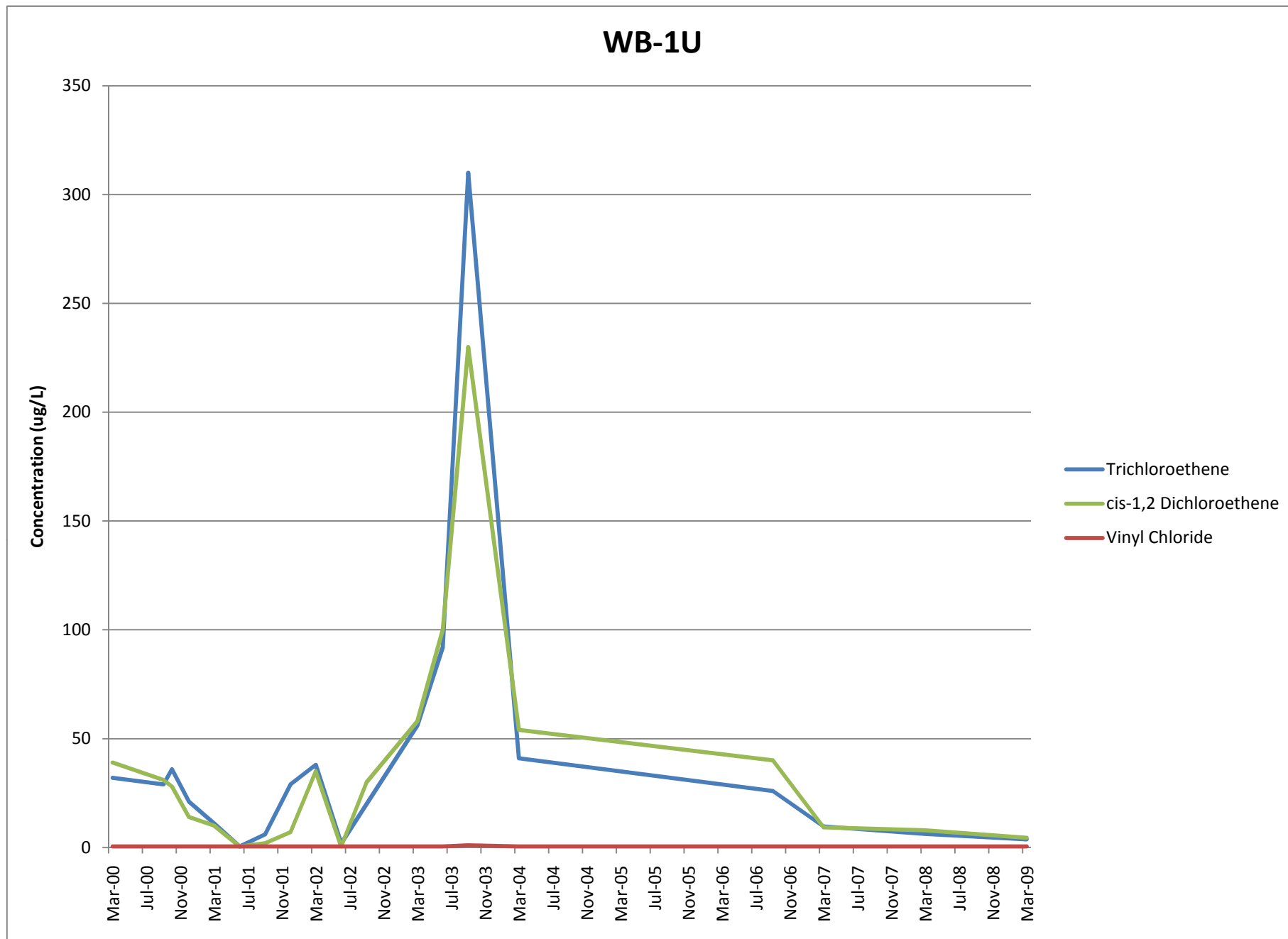
Index of VOC Concentration Trend Analysis Charts  
Hewlett-Packard Voluntary Remediation Project  
San German, Puerto Rico

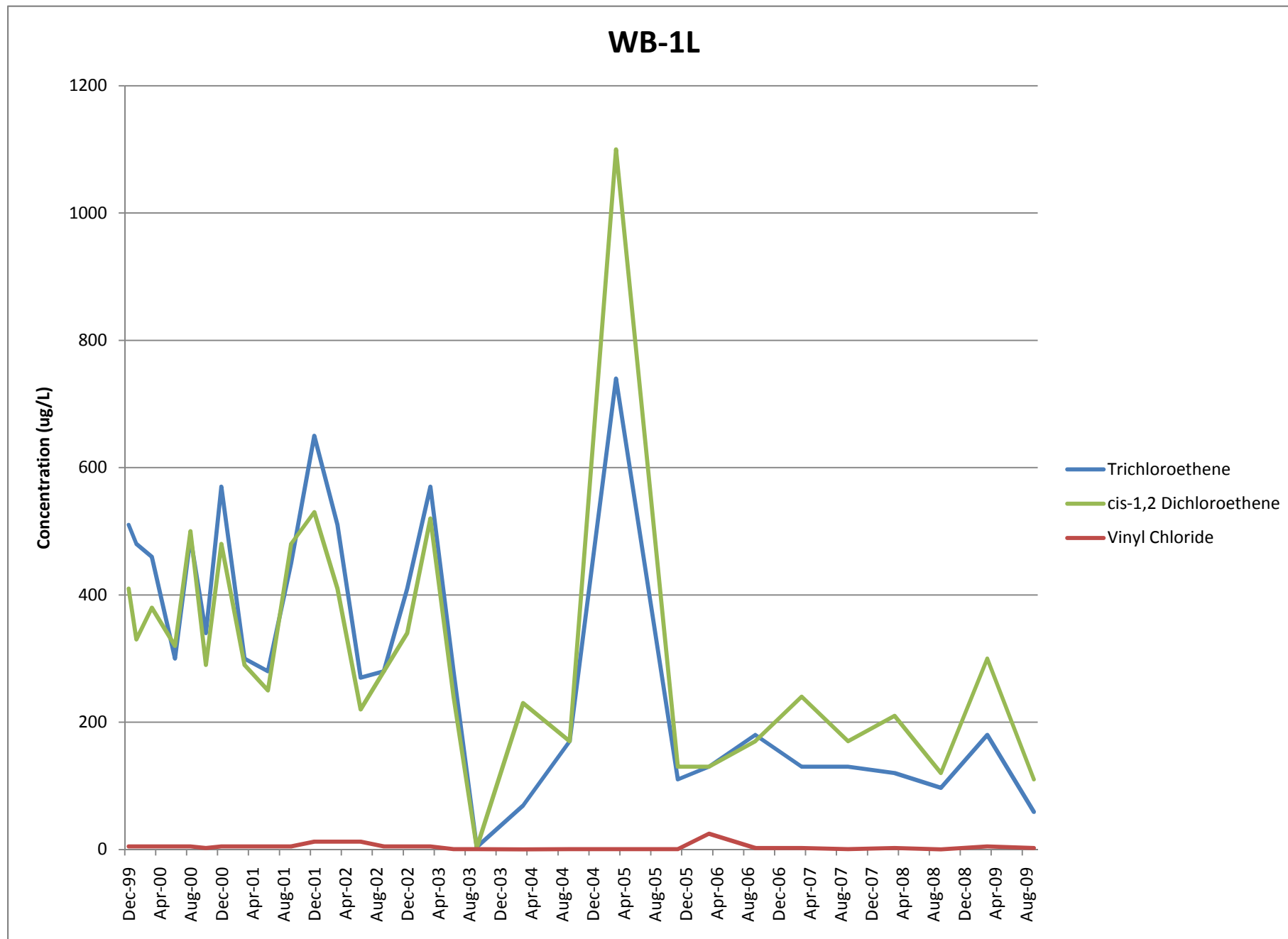
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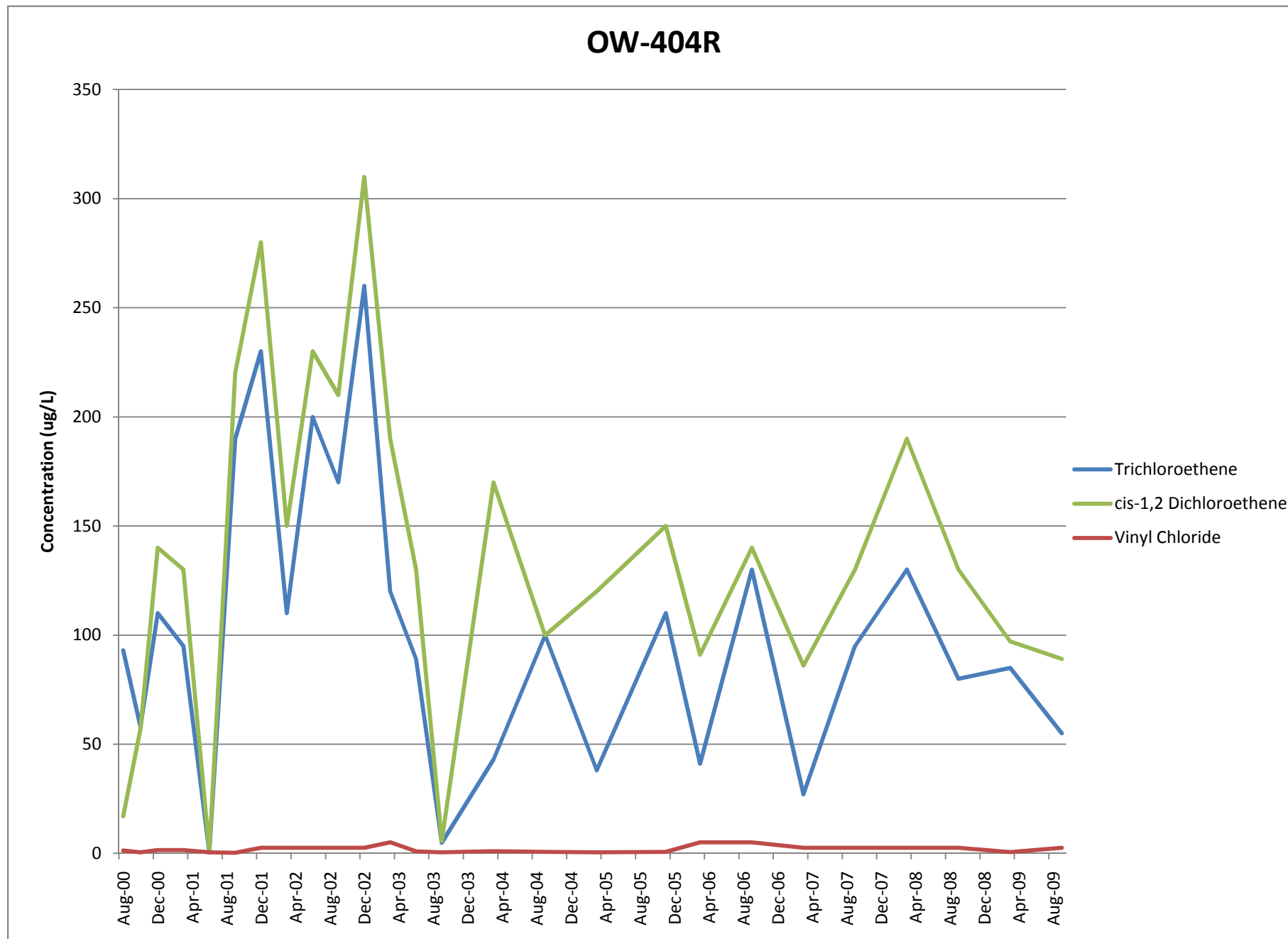
WB-1U: Screened in the Fill Unit.....	Page 1
WB-1L: Screened in the Saprolite Unit .....	Page 2
OW-404R: Screened in the Bedrock Unit .....	Page 3
OW-404L: Screened in the Saprolite Unit.....	Page 4
GZ-502L: Screened in the Saprolite Unit.....	Page 5
WB-2L: Screened in the Saprolite Unit.....	Page 6
WB-4L: Screened in the Saprolite Unit.....	Page 7
GZ-515U: Screened in the Alluvium Unit.....	Page 8
OW-305U: Screened in the Fill Unit.....	Page 9
OW-101: Screened in the Fill Unit.....	Page 10
GZ-506R: Screened in the Bedrock Unit.....	Page 11
OW-304R: Screened in the Bedrock Unit.....	Page 12
OW-304L: Screened in the Saprolite Unit.....	Page 13
GZ-504R: Screened in the Bedrock Unit.....	Page 14
GZ-505L: Screened in the Saprolite Unit.....	Page 15
GZ-505R: Screened in the Bedrock Unit.....	Page 16
OW-301: Screened in the Saprolite Unit.....	Page 17
OW-404U: Screened in the Fill Unit.....	Page 18

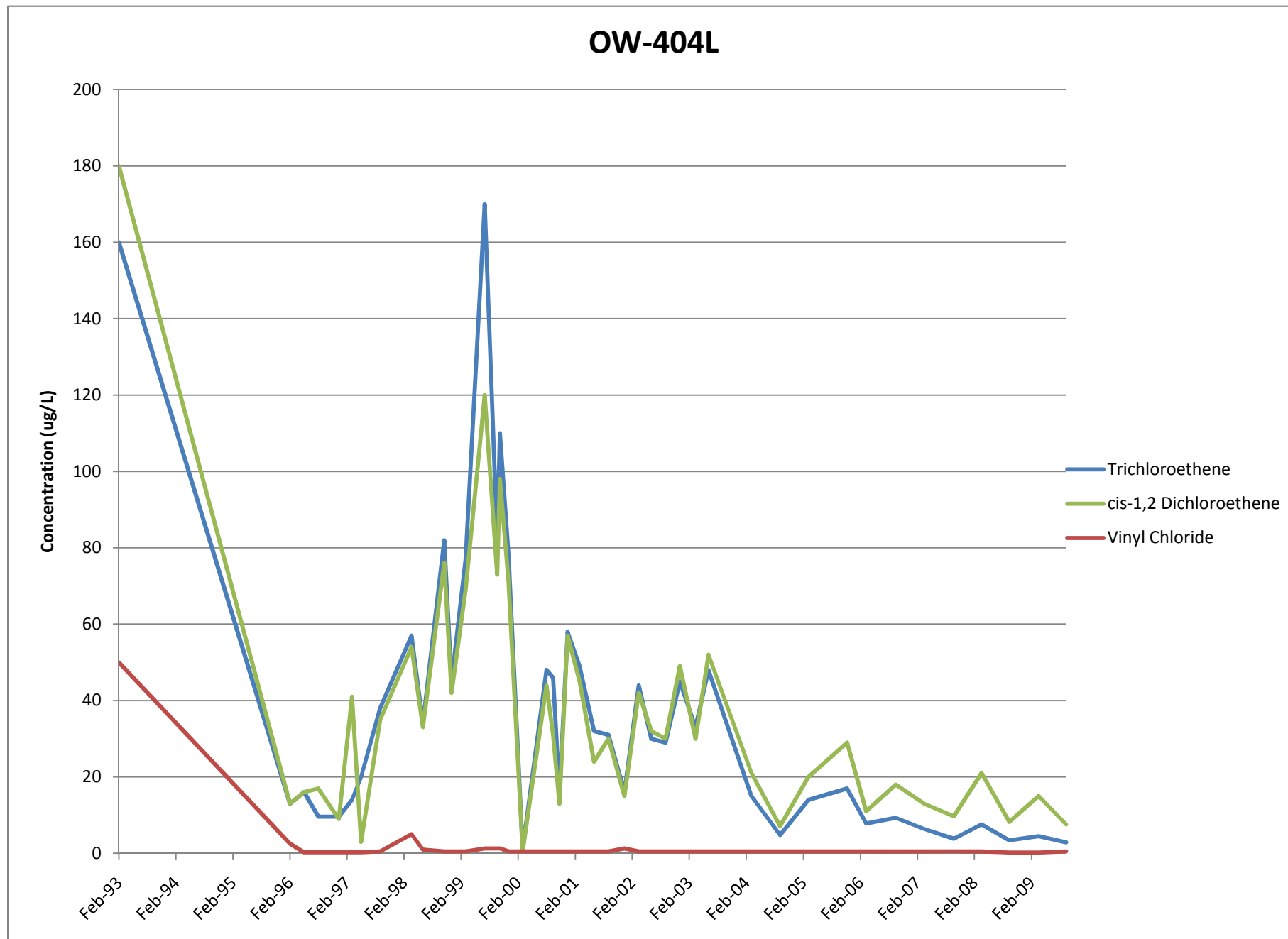
**Notes:**

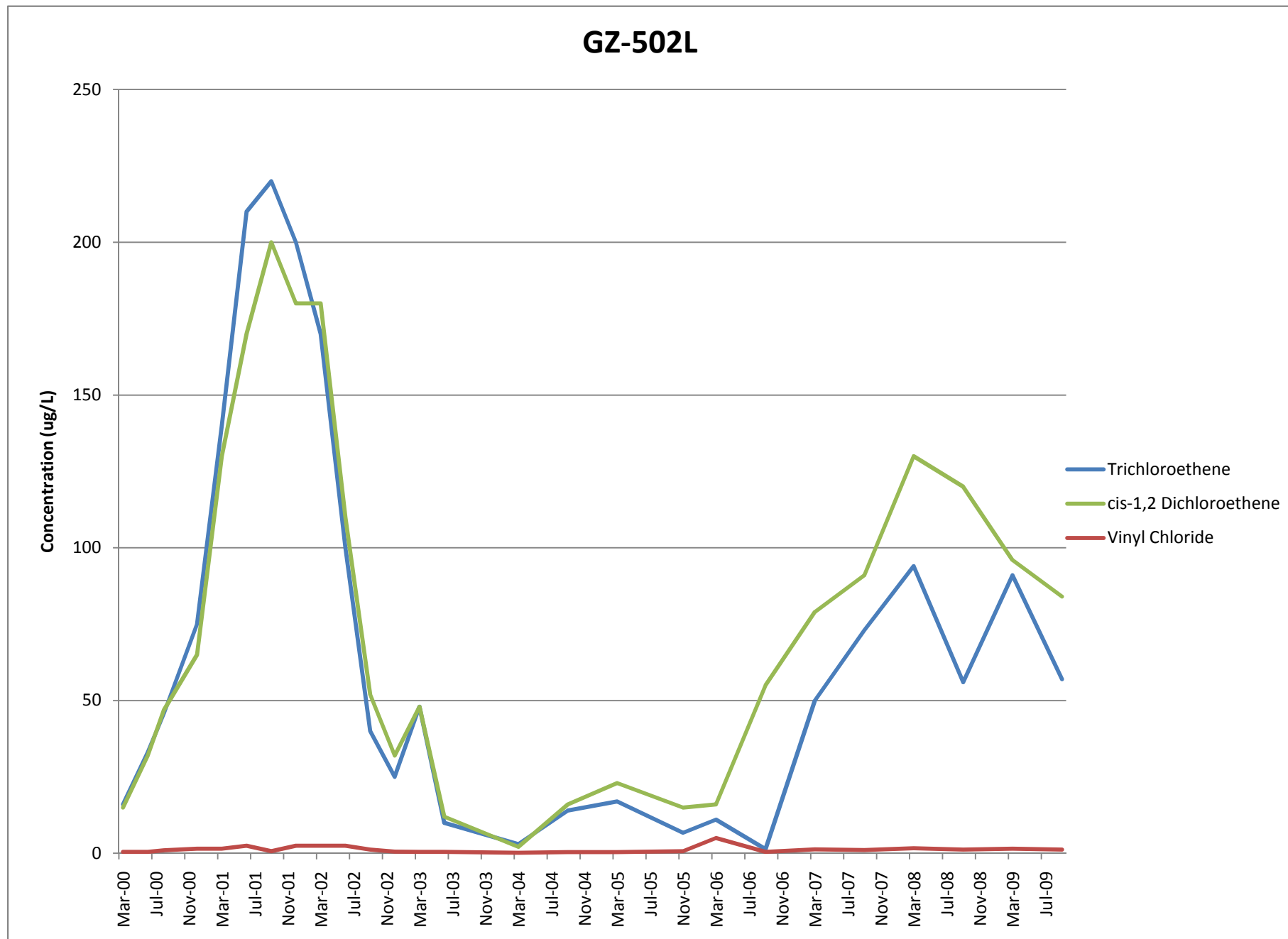
1. In instances where a constituent was not detected, half of the reporting limit was used as the concentration.
2. Data that was reported with a qualifier was treated as if it was not reported with a qualifier in this analysis. In general, this led to a more conservative analysis.
3. VOC = Volatile Organic Compounds
4. For several wells (WB-1L, OW-404R, OW-404L, WB-2L, WB-4L, GZ-515U, GZ-506R, GZ-304L, and OW-301) the vinyl chloride analysis was based exclusively on non-detect and qualified results. For three of those wells (WB-4L, GZ-515U, and OW-301) this is true for the trichloroethene and the cis-1,2-dichloroethene analyses as well. For two wells (OW-101 and OW-304R), many of the reporting limits were very high. For OW-101, any of the values that are greater than 15 ug/L are based on non-detects. For OW-304R, any of the values that are greater than 110 ug/L are based on non-detects.
5. In all previous reports, results for OW-305U have been reported as results for OW-305I, and results for OW-305I have been reported as results for OW-305U. This has been corrected for this and subsequent reports.



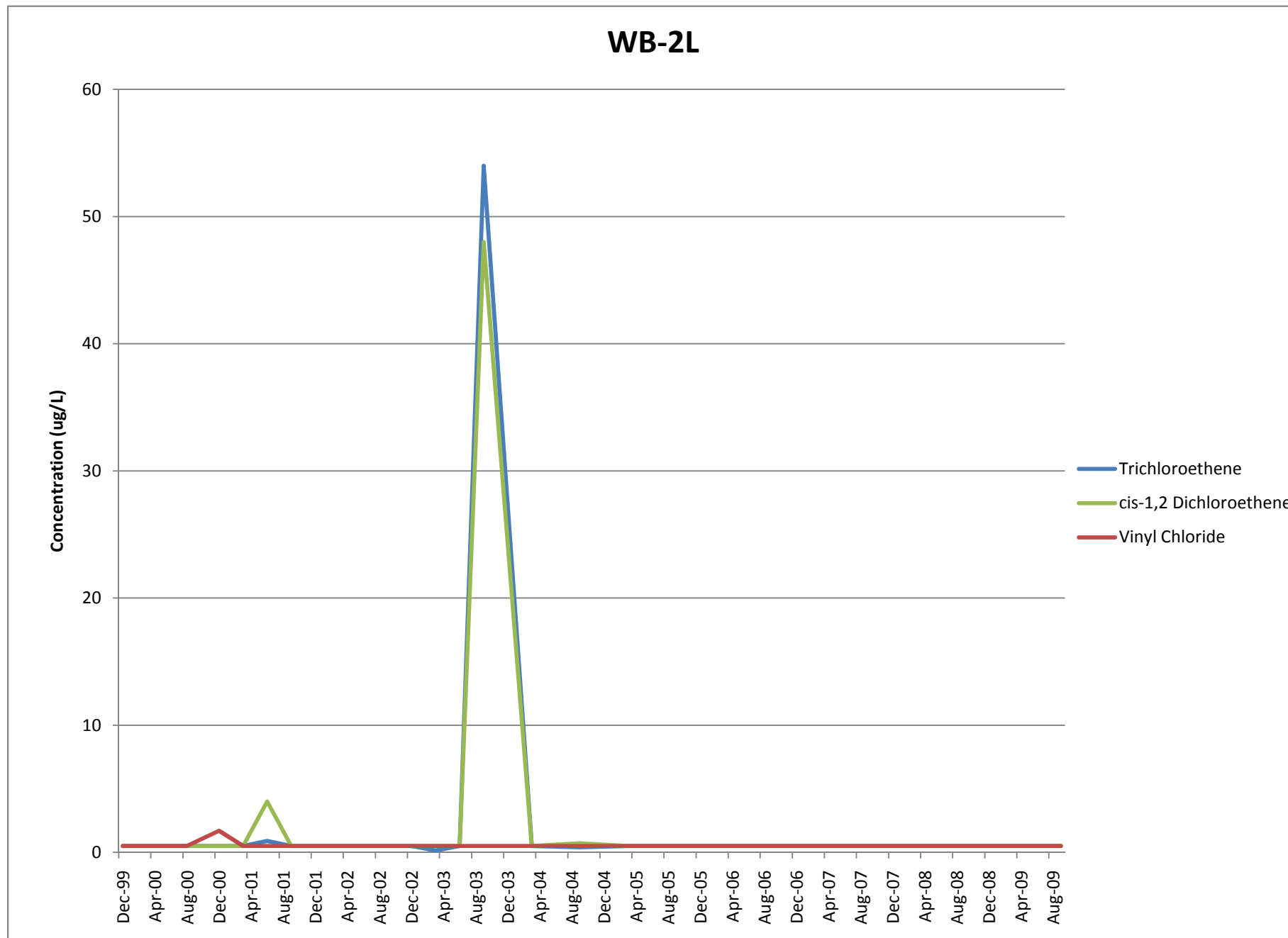


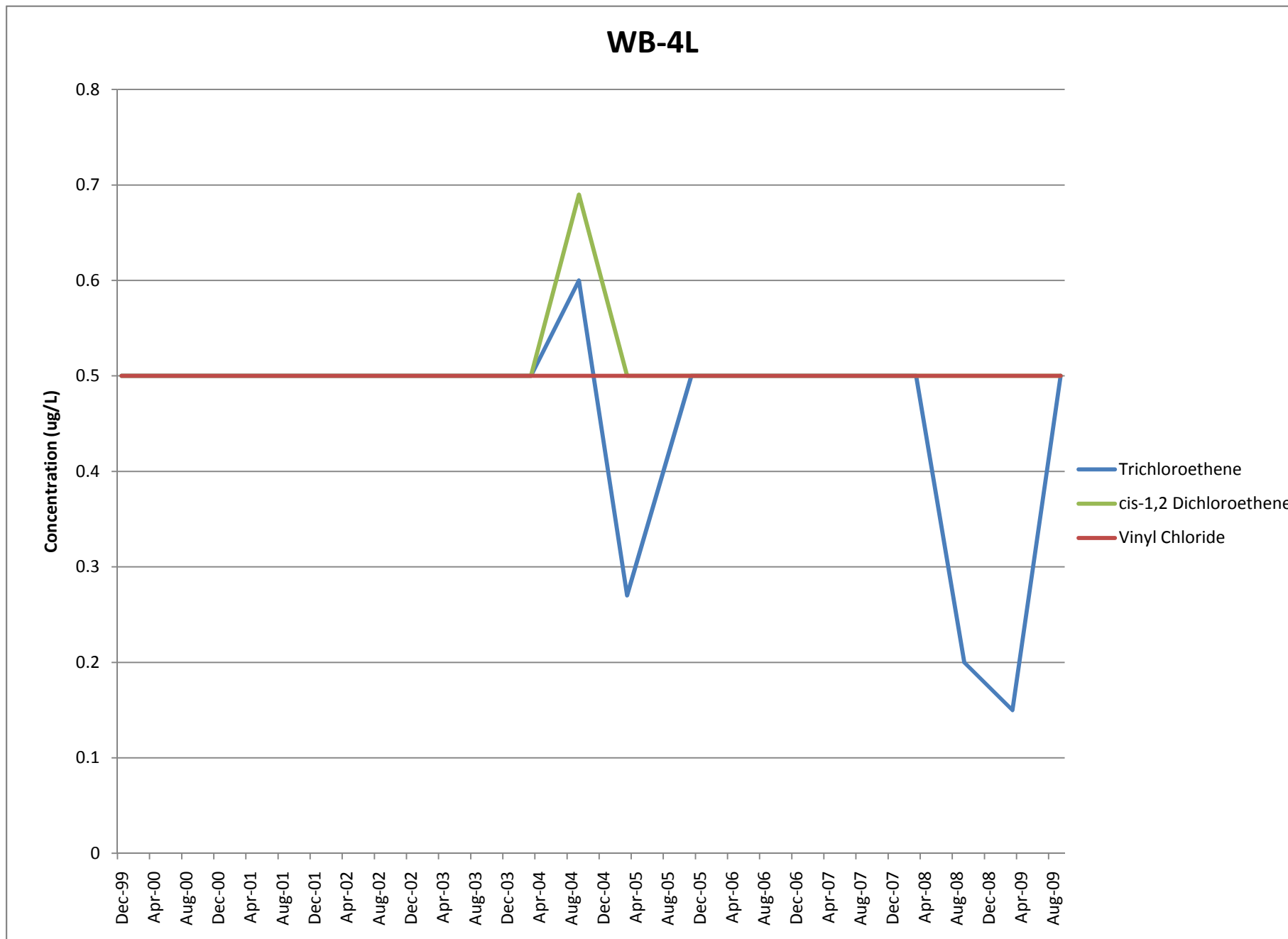


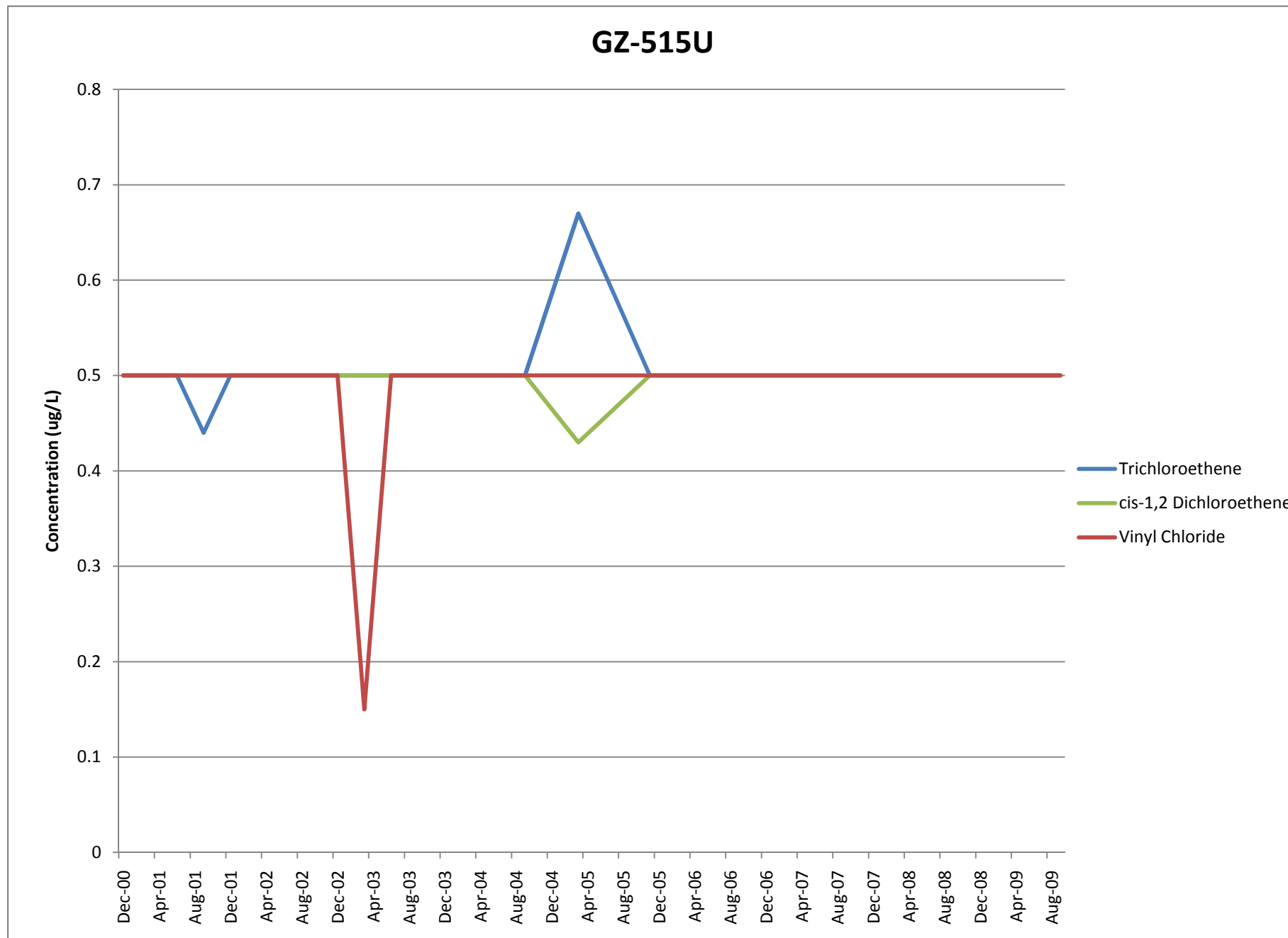


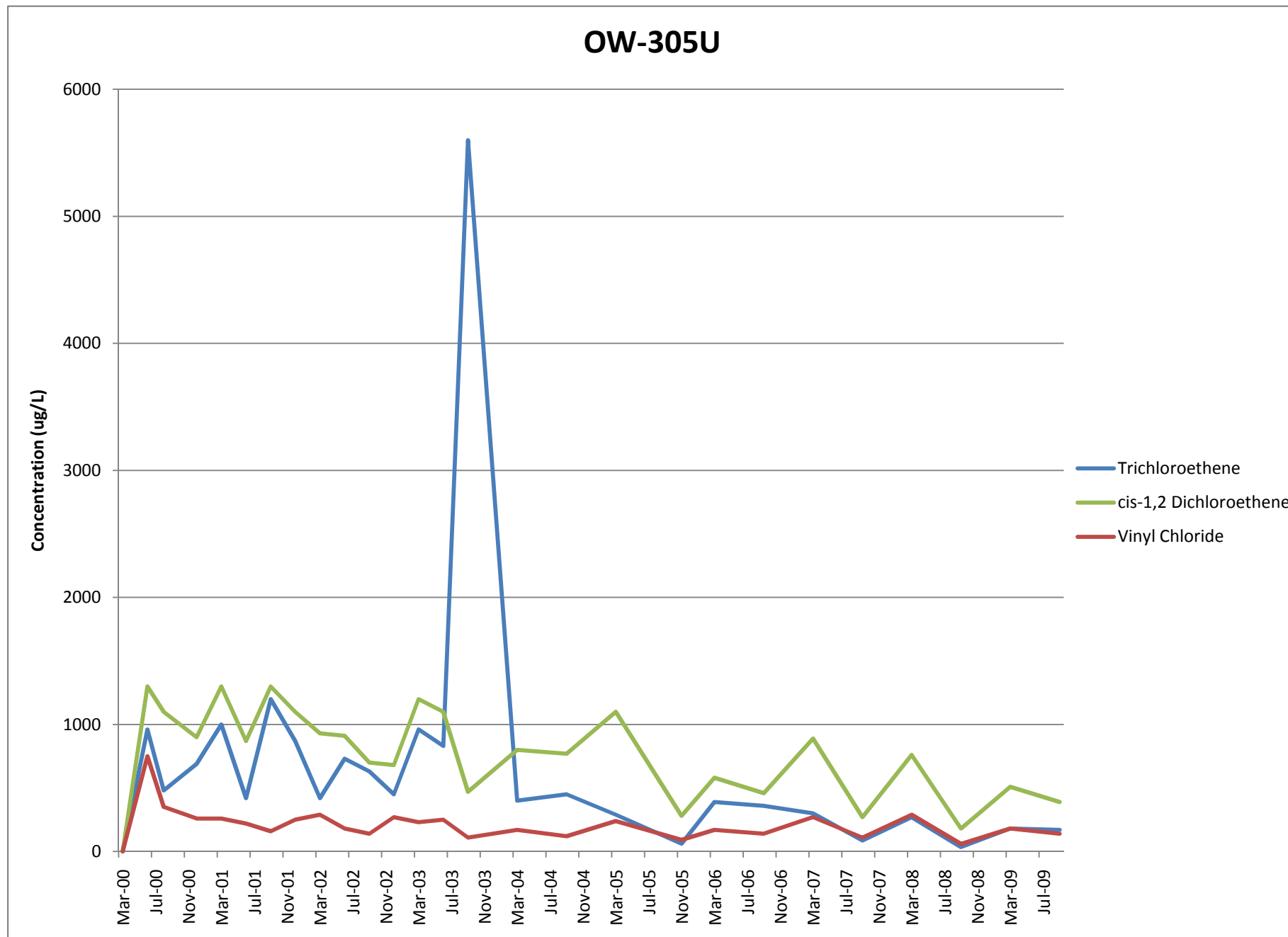


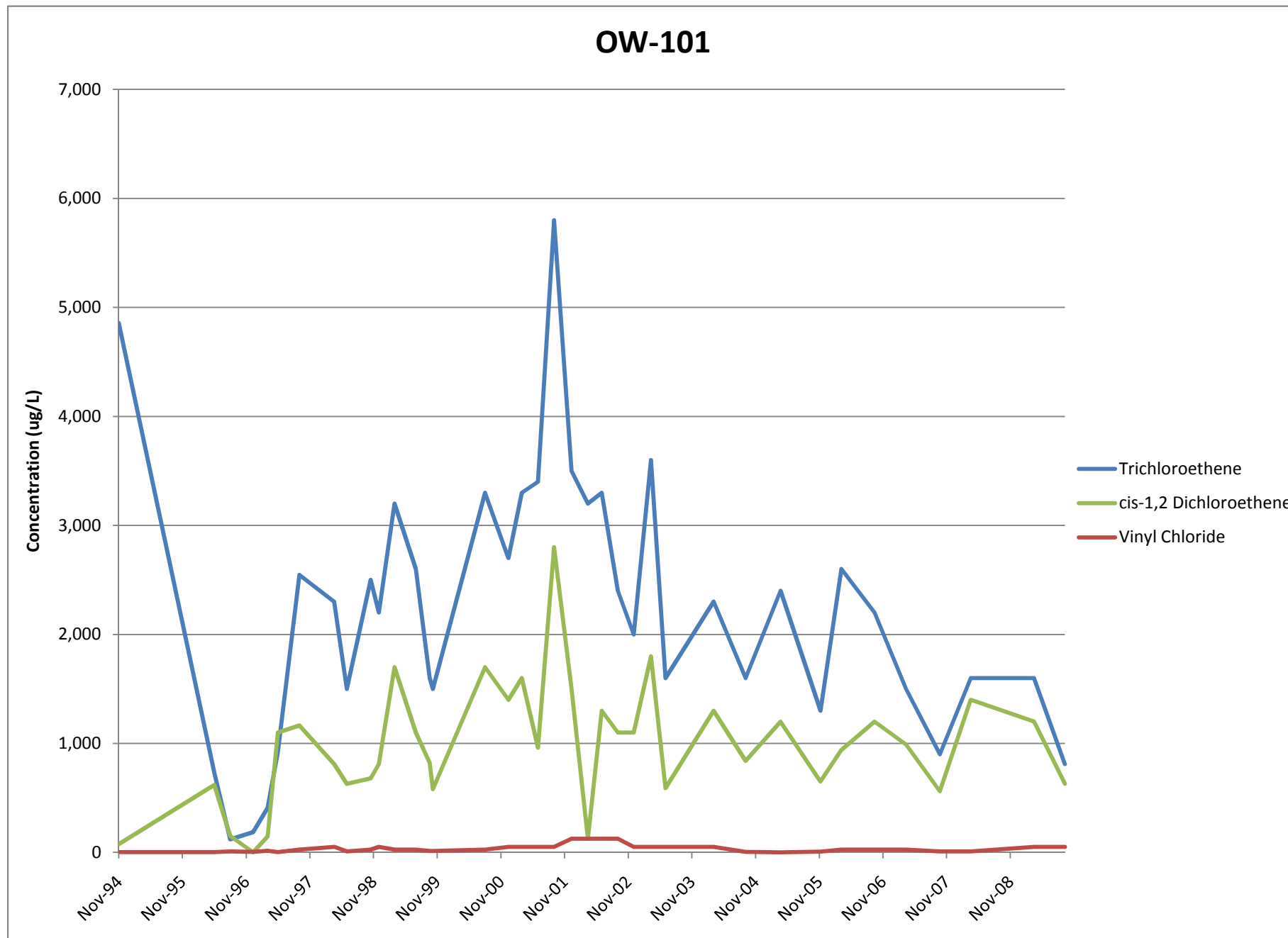


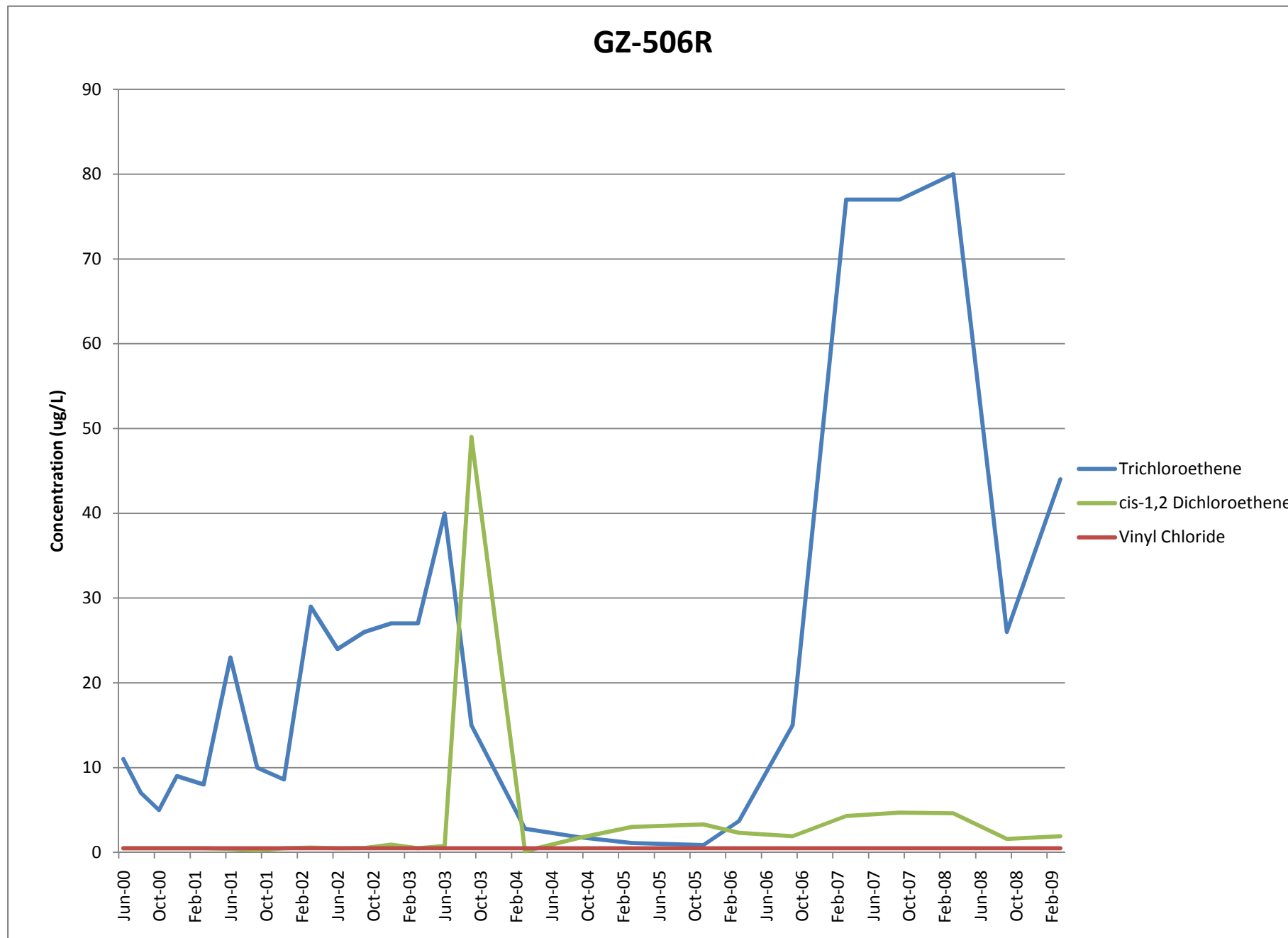


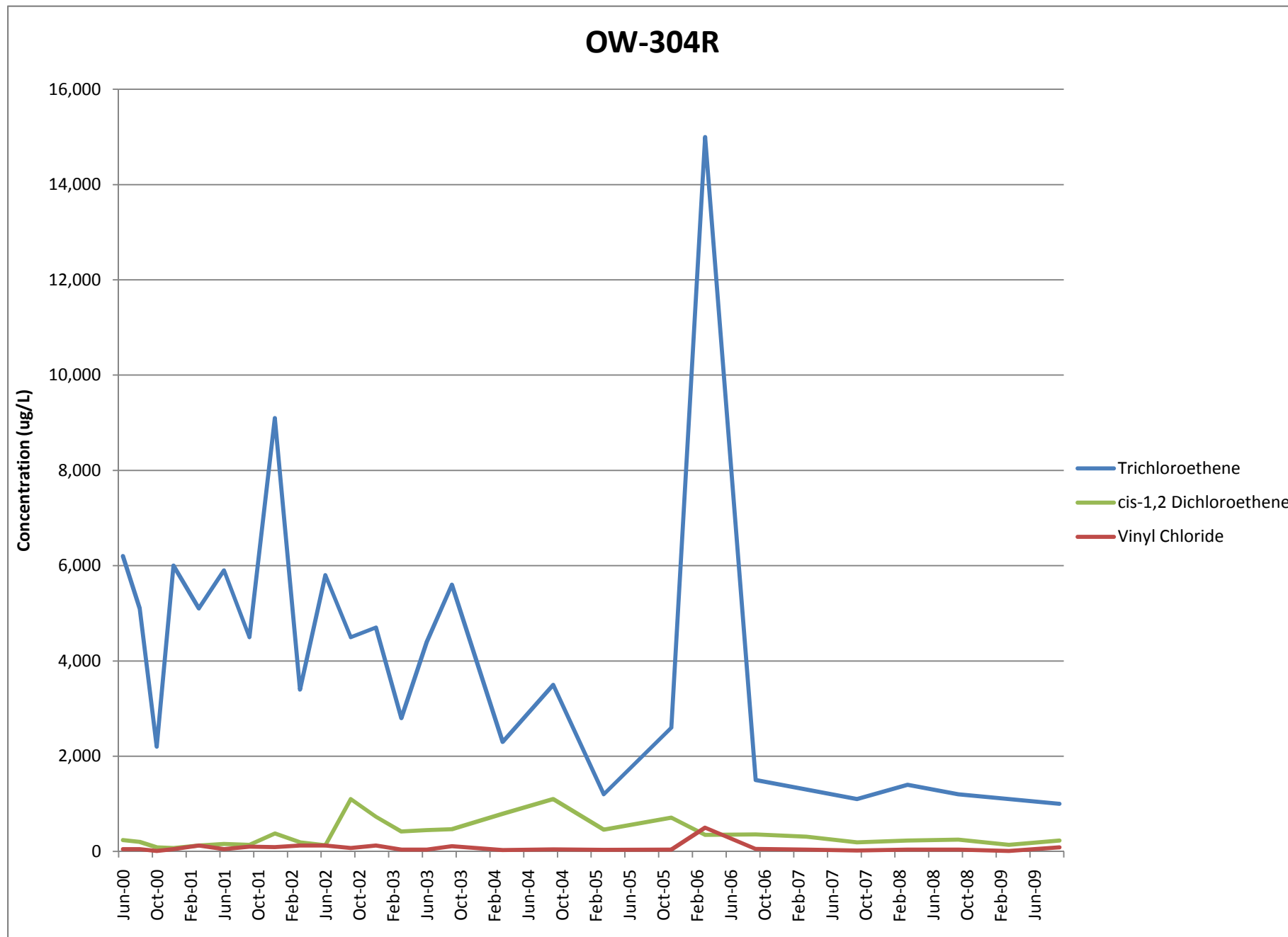


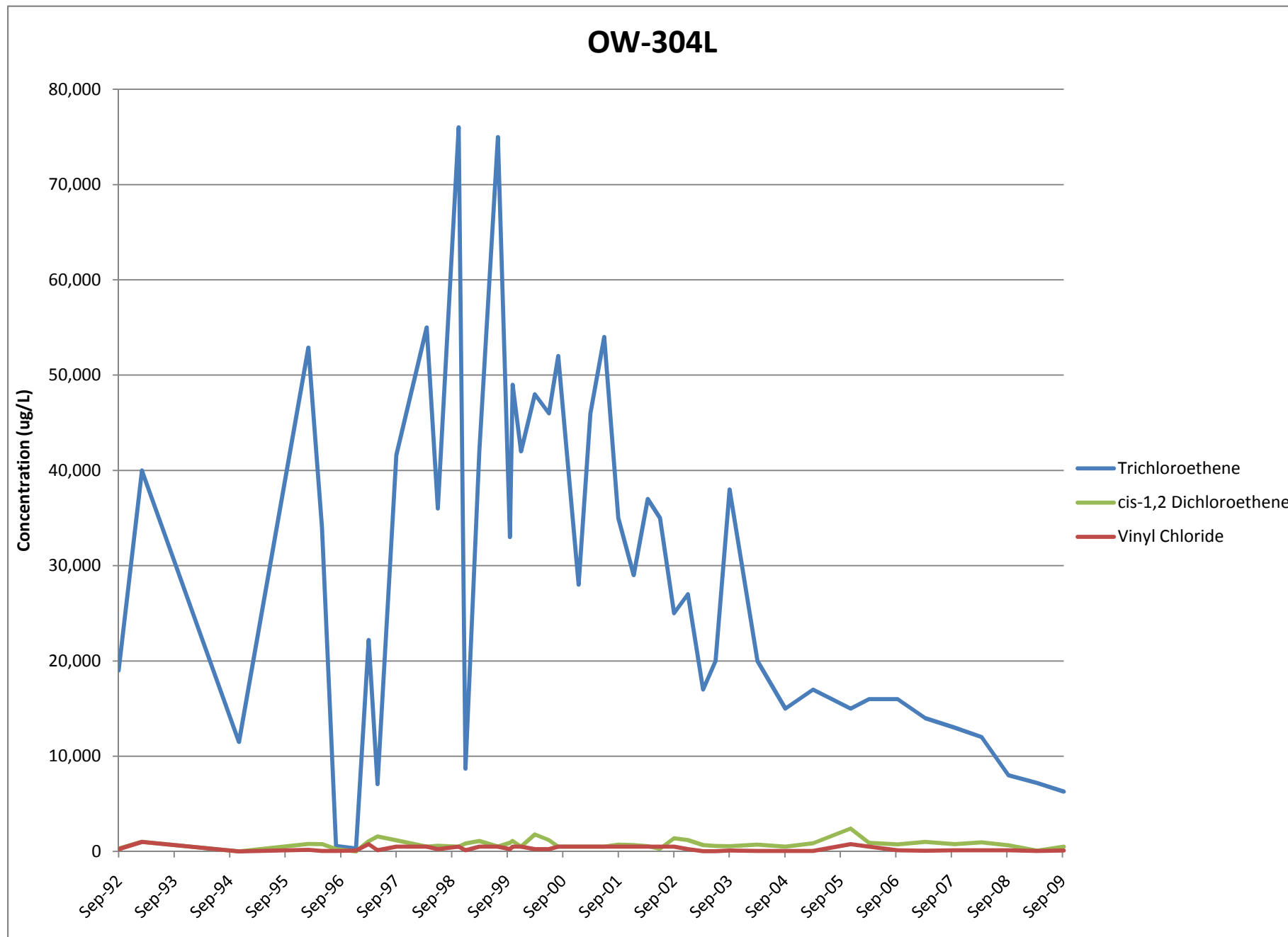




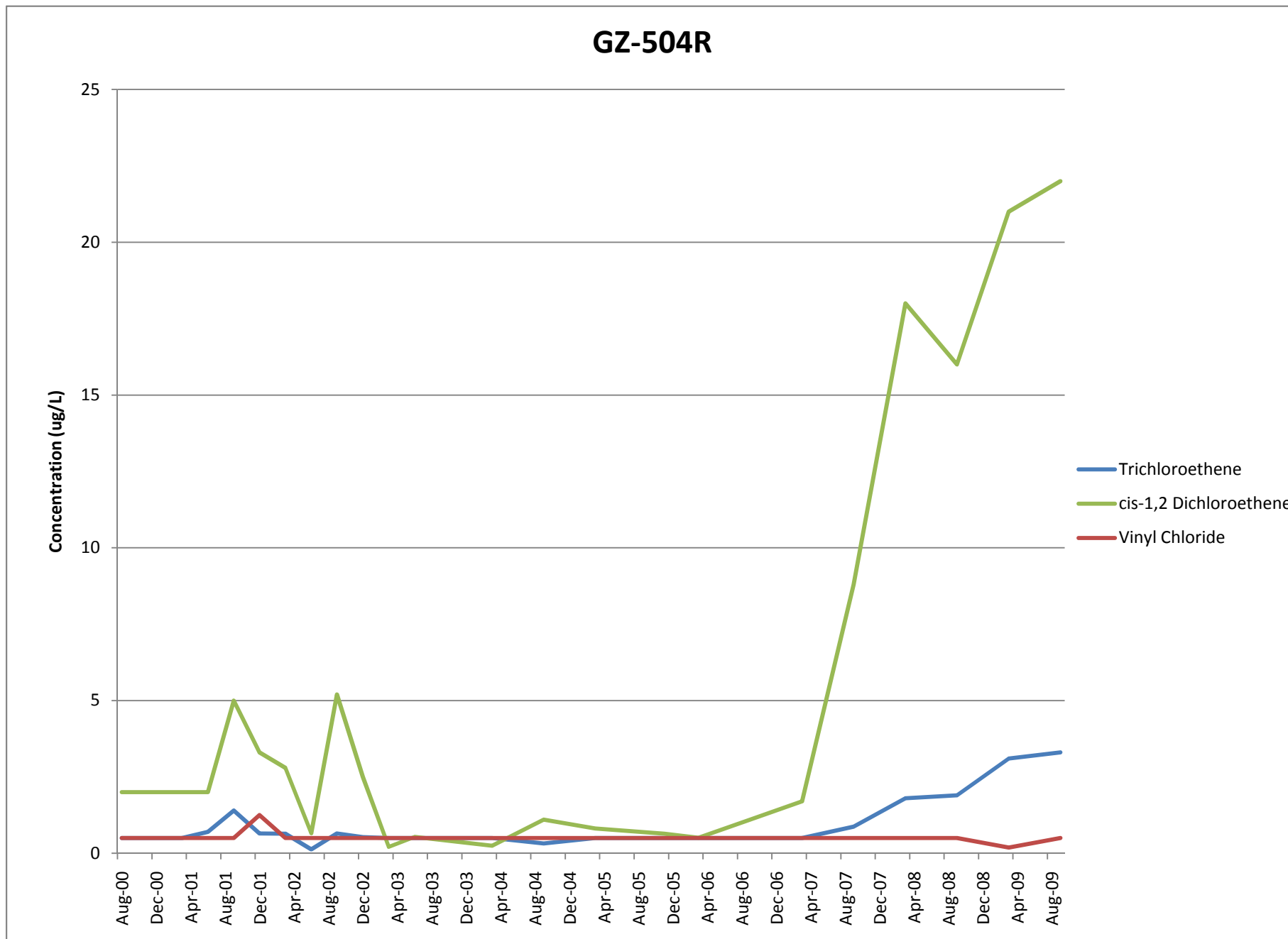


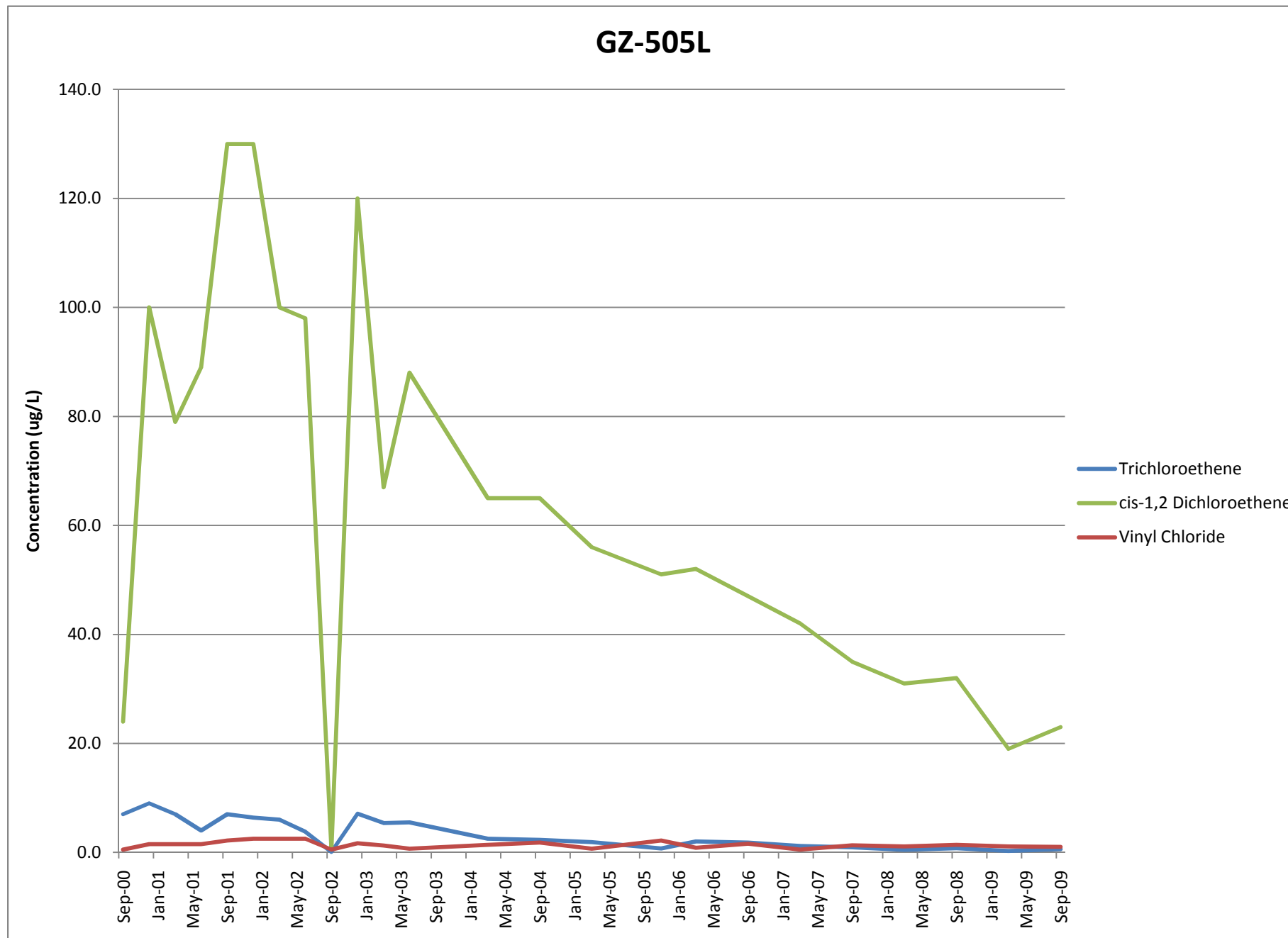


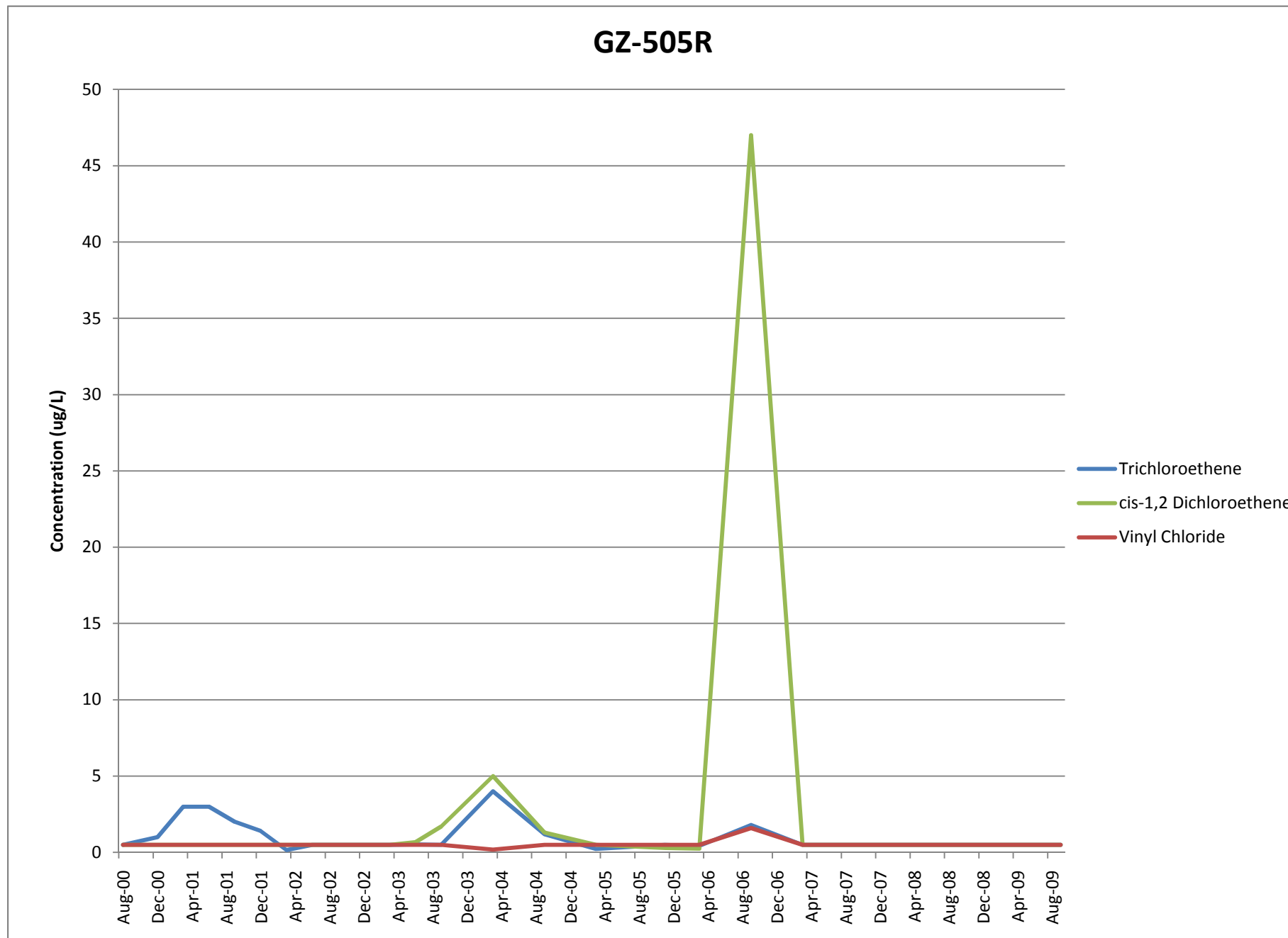


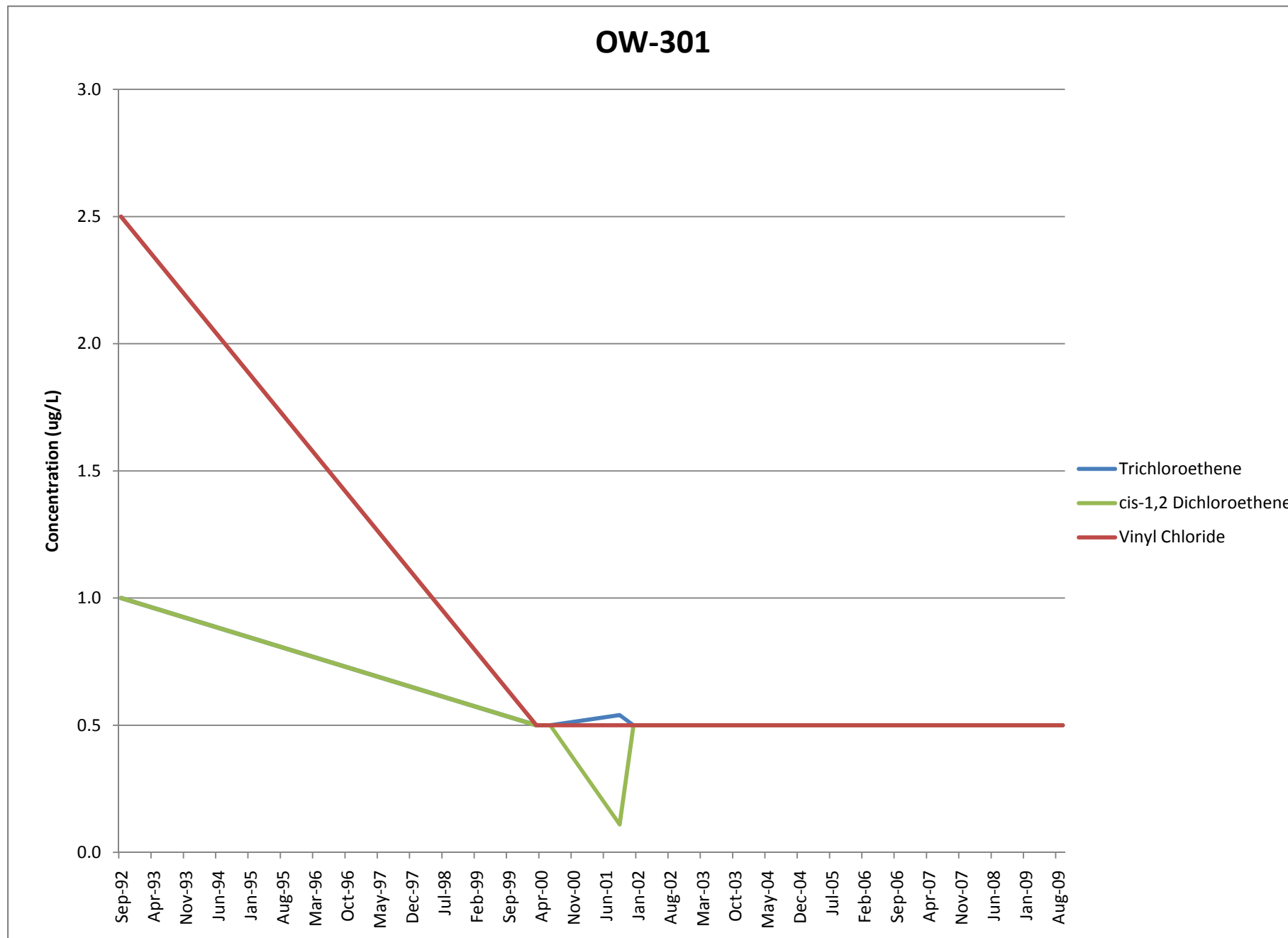


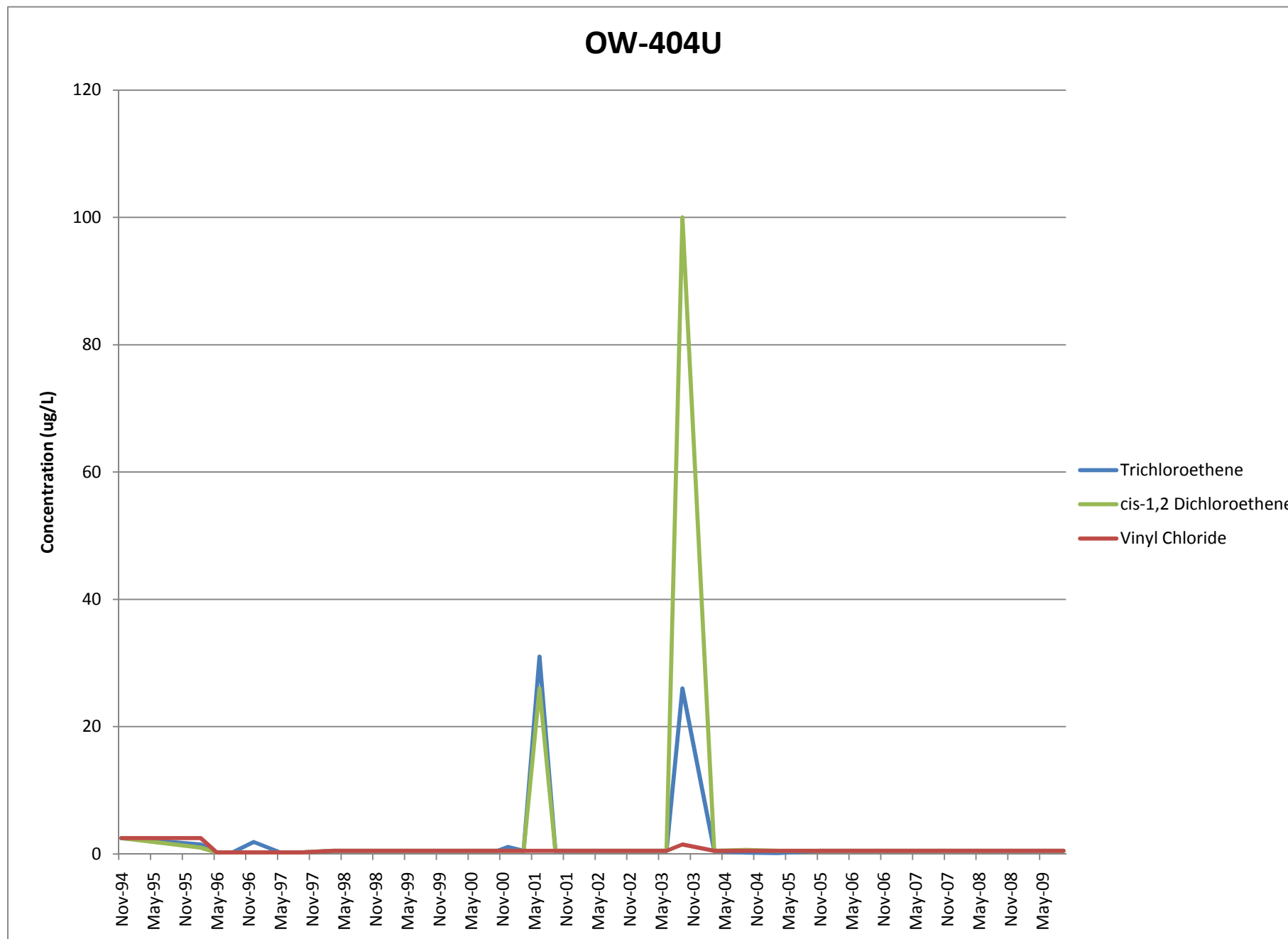












## **APPENDIX C**

### **VOC MOLARITY TREND ANALYSIS CHARTS**

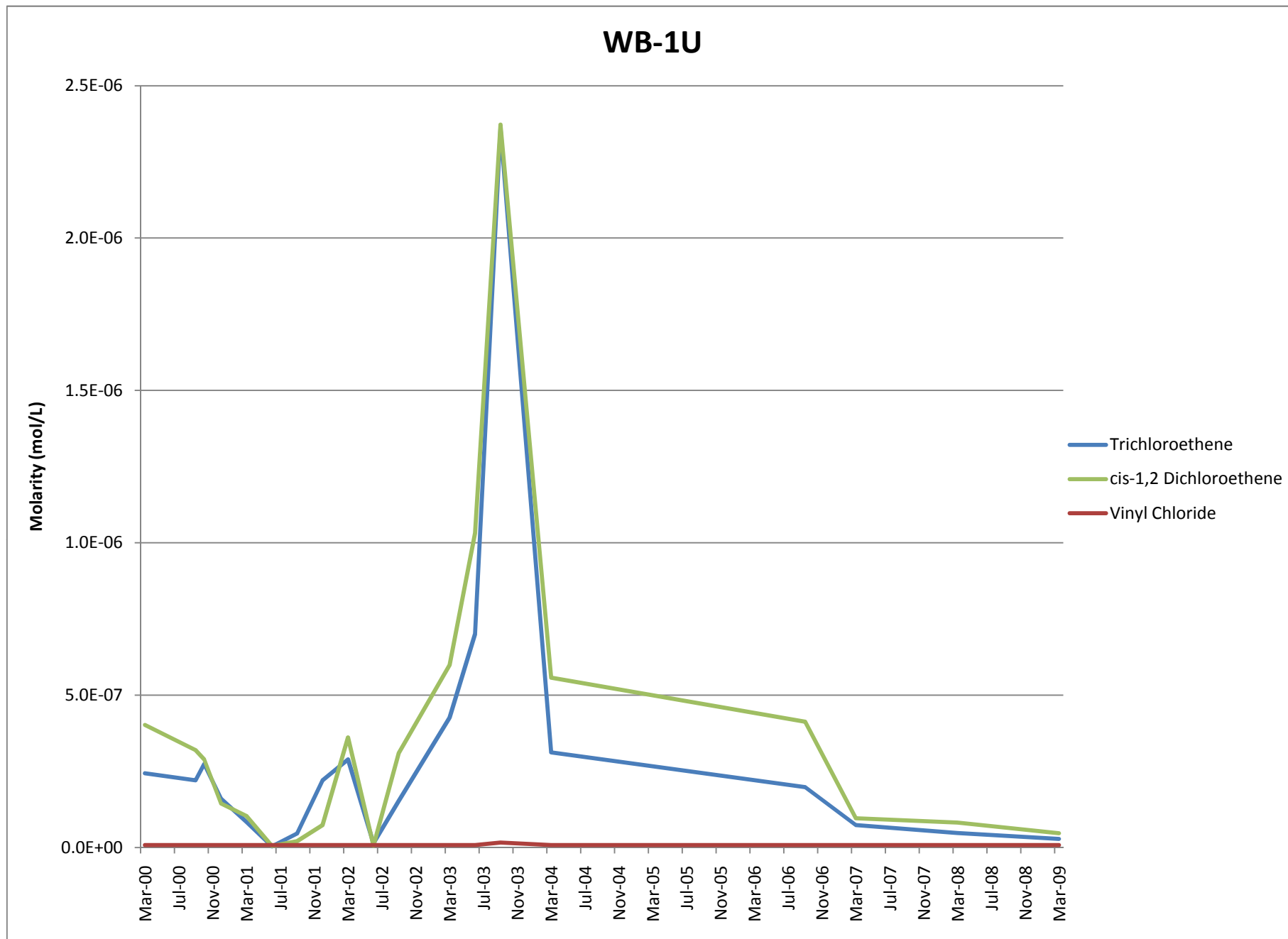
Index of VOC Molarity Trend Analysis Charts  
Hewlett-Packard Voluntary Remediation Project  
San German, Puerto Rico

File No. 01.0024065.10

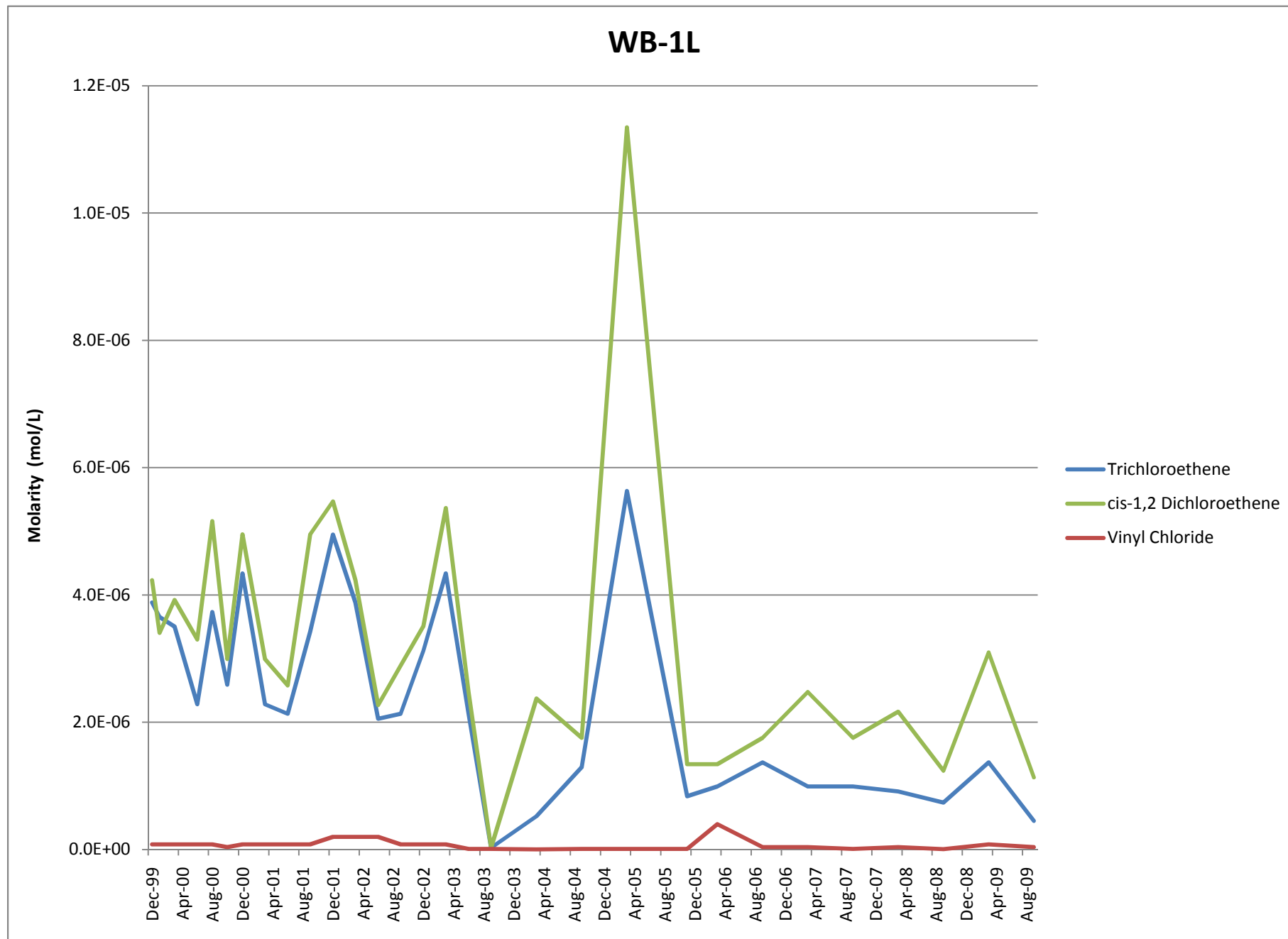
WB-1U: Screened in the Fill Unit.....	Page 1
WB-1L: Screened in the Saprolite Unit .....	Page 2
OW-404R: Screened in the Bedrock Unit .....	Page 3
OW-404L: Screened in the Saprolite Unit.....	Page 4
GZ-502L: Screened in the Saprolite Unit.....	Page 5
WB-2L: Screened in the Saprolite Unit.....	Page 6
WB-4L: Screened in the Saprolite Unit.....	Page 7
GZ-515U: Screened in the Alluvium Unit.....	Page 8
OW-305U: Screened in the Fill Unit.....	Page 9
OW-101: Screened in the Fill Unit.....	Page 10
GZ-506R: Screened in the Bedrock Unit.....	Page 11
OW-304R: Screened in the Bedrock Unit.....	Page 12
OW-304L: Screened in the Saprolite Unit.....	Page 13
GZ-504R: Screened in the Bedrock Unit.....	Page 14
GZ-505L: Screened in the Saprolite Unit.....	Page 15
GZ-505R: Screened in the Bedrock Unit.....	Page 16
OW-301: Screened in the Saprolite Unit.....	Page 17
OW-404U: Screened in the Fill Unit.....	Page 18

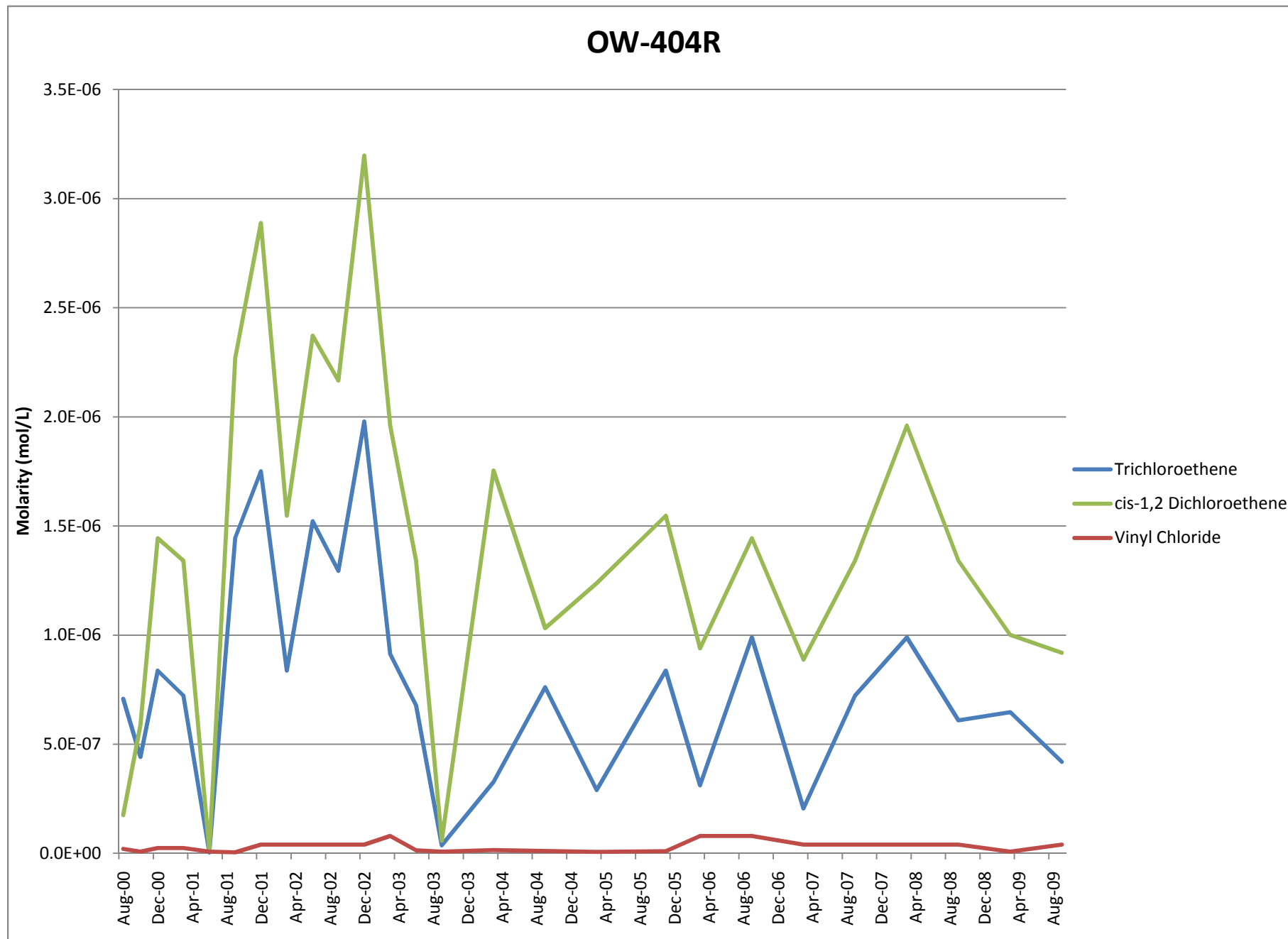
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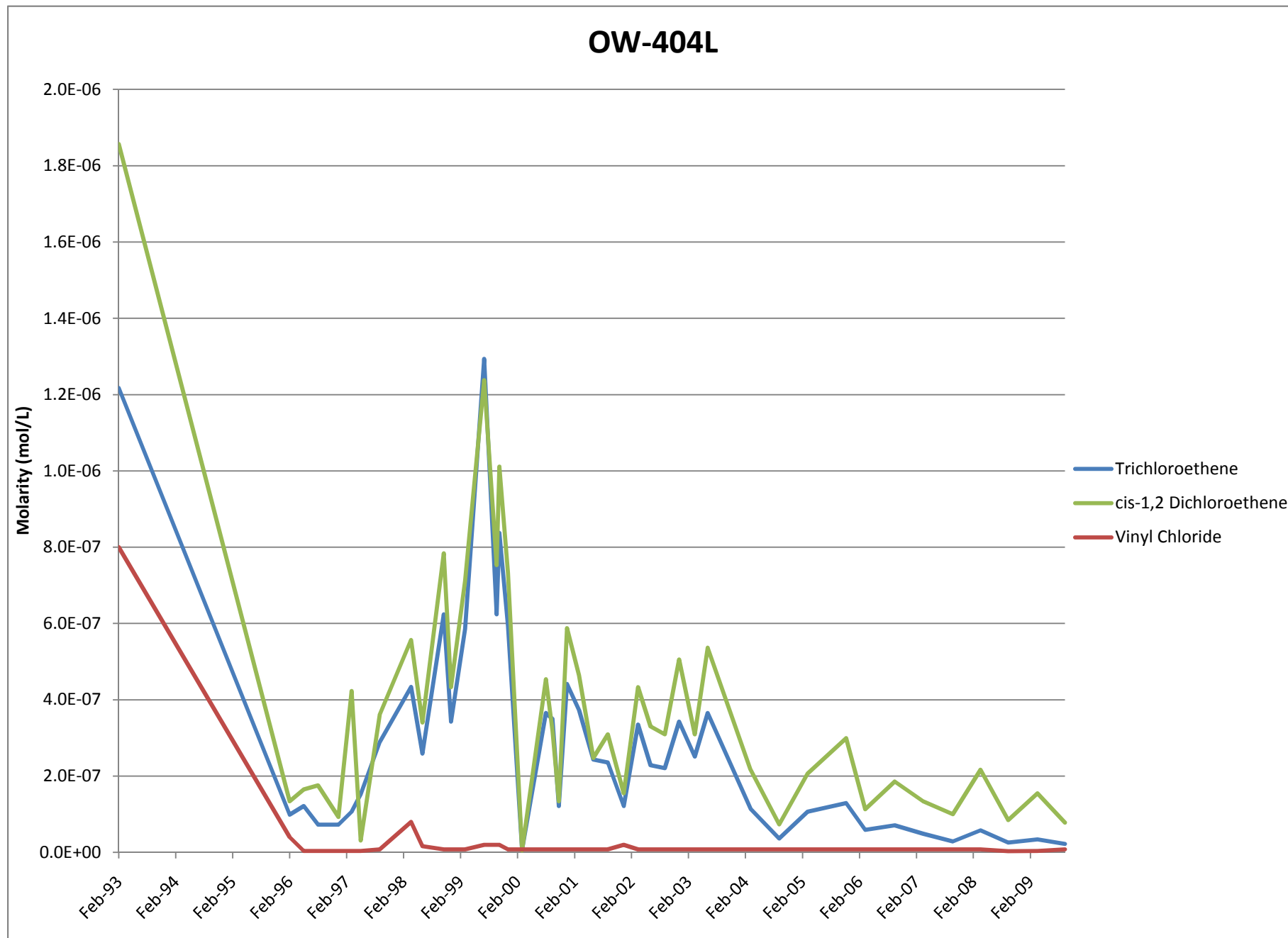
1. In instances where a constituent was not detected, half of the reporting limit was used as the molarity.
2. Data that was reported with a qualifier was treated as if it was not reported with a qualifier in this analysis. In general, this led to a more conservative analysis.
3. VOC = Volatile Organic Compounds; TCE = Trichloroethene; DCE = cis-1,2-Dichloroethene; VC = Vinyl Chloride
4. For several wells (WB-1L, OW-404R, OW-404L, WB-2L, WB-4L, GZ-515U, GZ-506R, GZ-304L, and OW-301) the vinyl chloride analysis was based exclusively on non-detect and qualified results. For three of those wells (WB-4L, GZ-515U, and OW-301) this is true for the trichloroethene and the cis-1,2-dichloroethene analyses as well. For two wells (OW-101 and OW-304R), many of the reporting limits were very high. For OW-101, any of the vinyl chloride values that are greater than 2.4E-7 mol/L are based on non-detects. For OW-304R, any of the vinyl chloride values that are greater than 2.0E-6 mol/L are based on non-detects.
5. In all previous reports, results for OW-305U have been reported as results for OW-305I, and results for OW-305I have been reported as results for OW-305U. This has been corrected for this and subsequent reports.
6. Molecular weights for the carbon (C – 12.01 g/mol), hydrogen (H – 1.01 g/mol), and chloride (Cl – 35.45 g/mol) atoms were used to calculate the molecular weights of TCE (C<sub>2</sub>HCl<sub>3</sub> – 131.38 g/mol), DCE (C<sub>2</sub>H<sub>2</sub>Cl<sub>2</sub> – 96.94 g/mol), and VC (C<sub>2</sub>H<sub>3</sub>Cl – 62.50 g/mol).
7. Molarity (in mol/L) was calculated by dividing the concentration (in ug/L\*10<sup>-6</sup> g/ug) by the molecular weight (in g/mol).

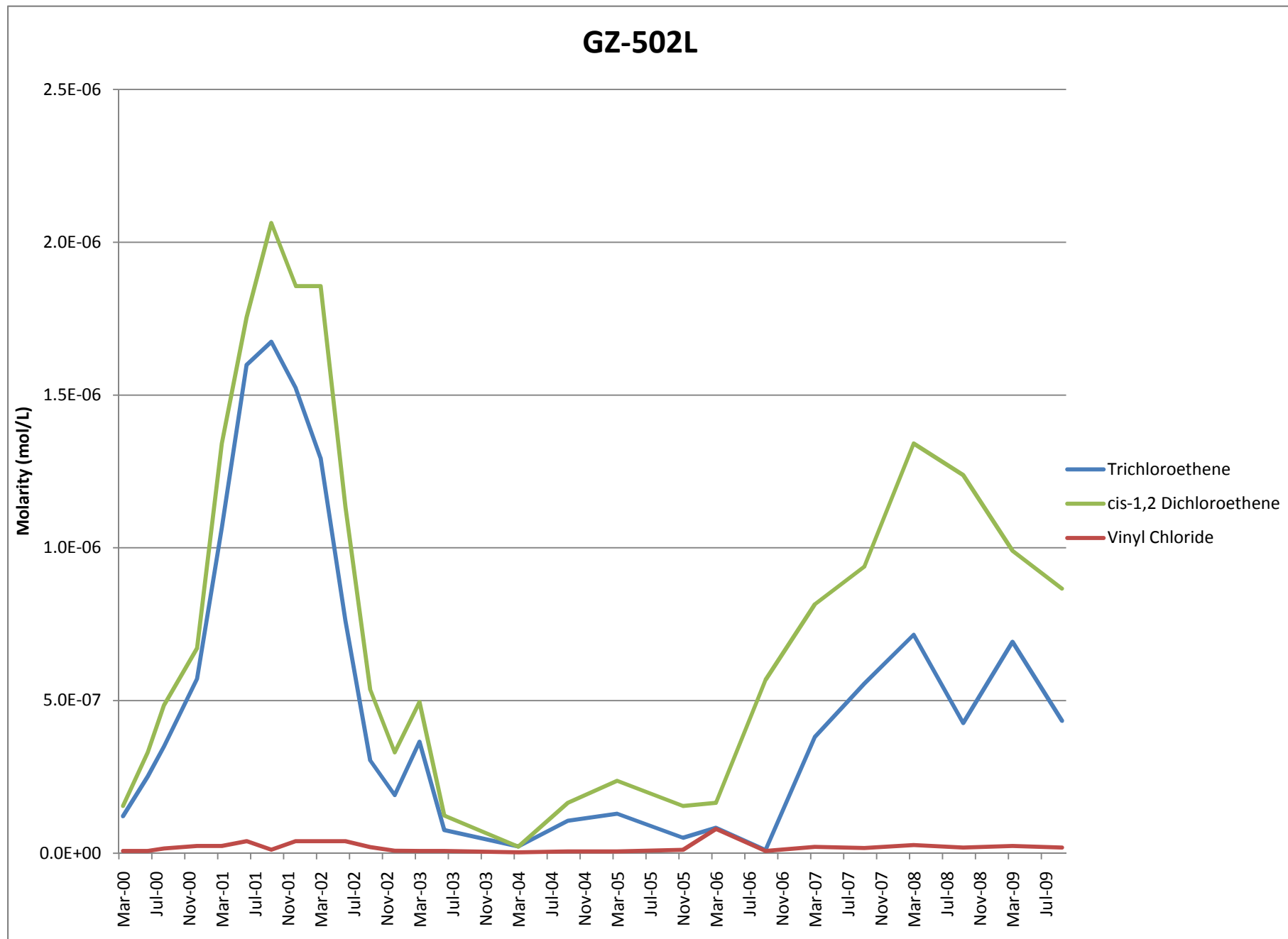


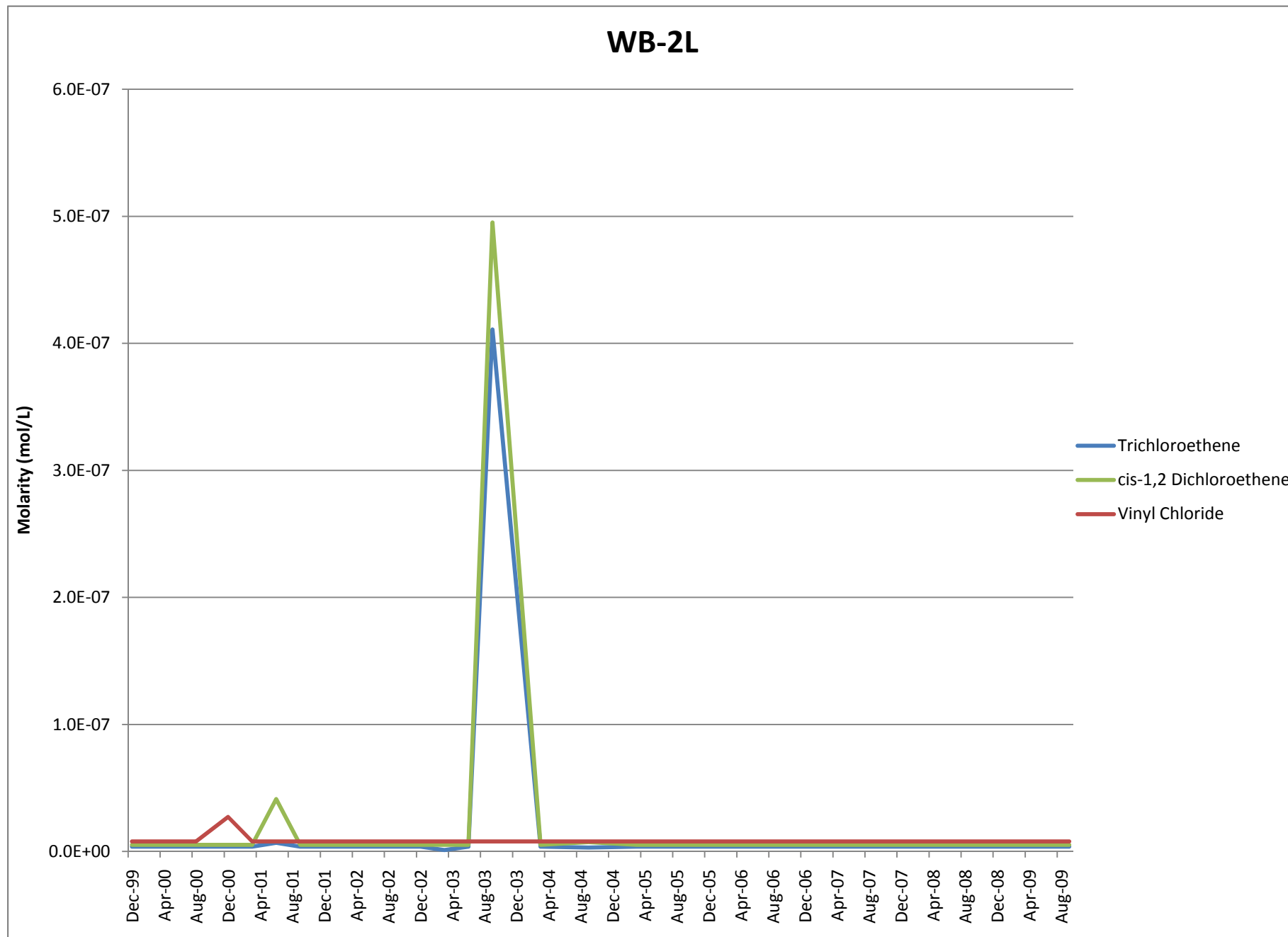


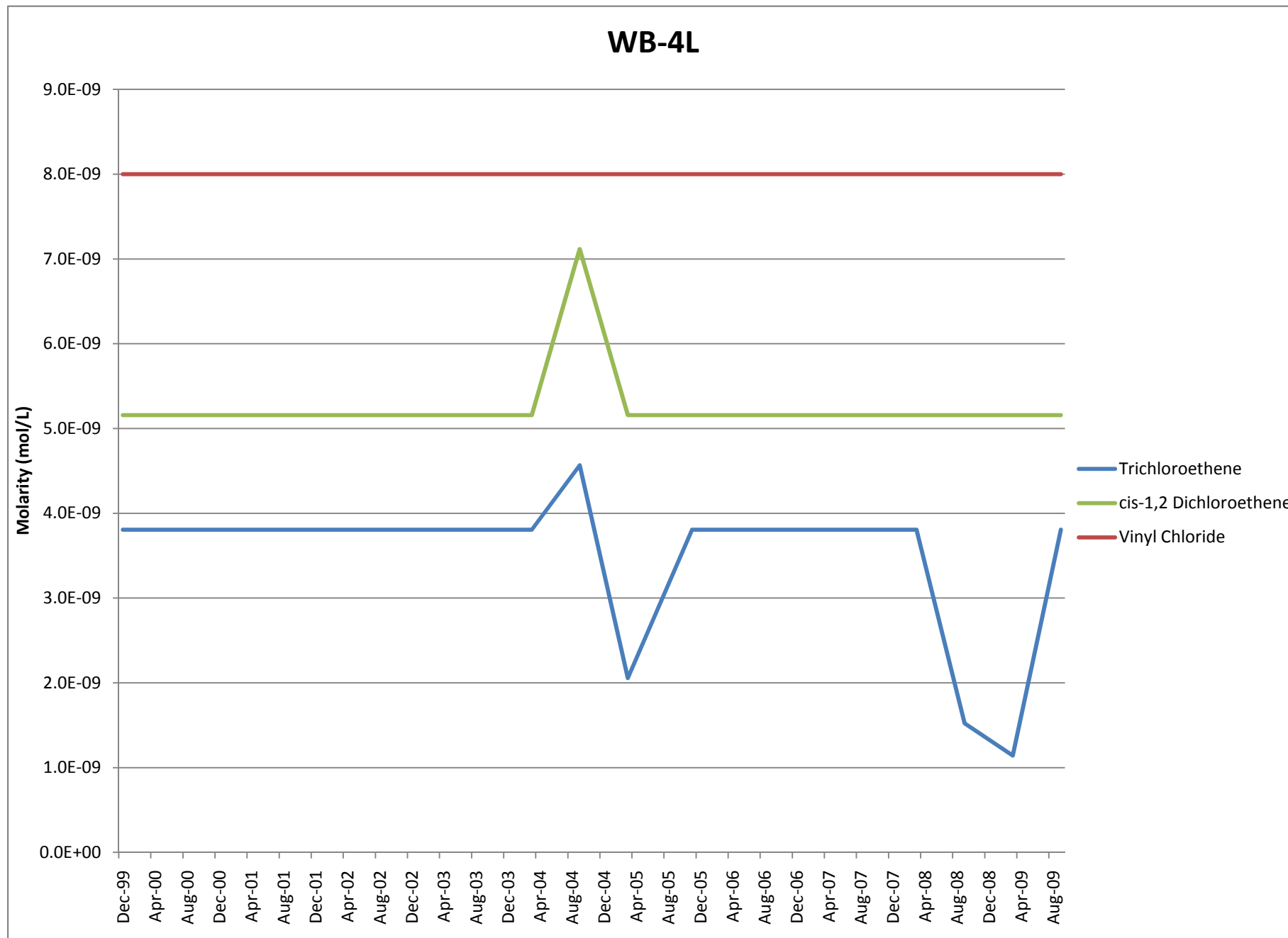


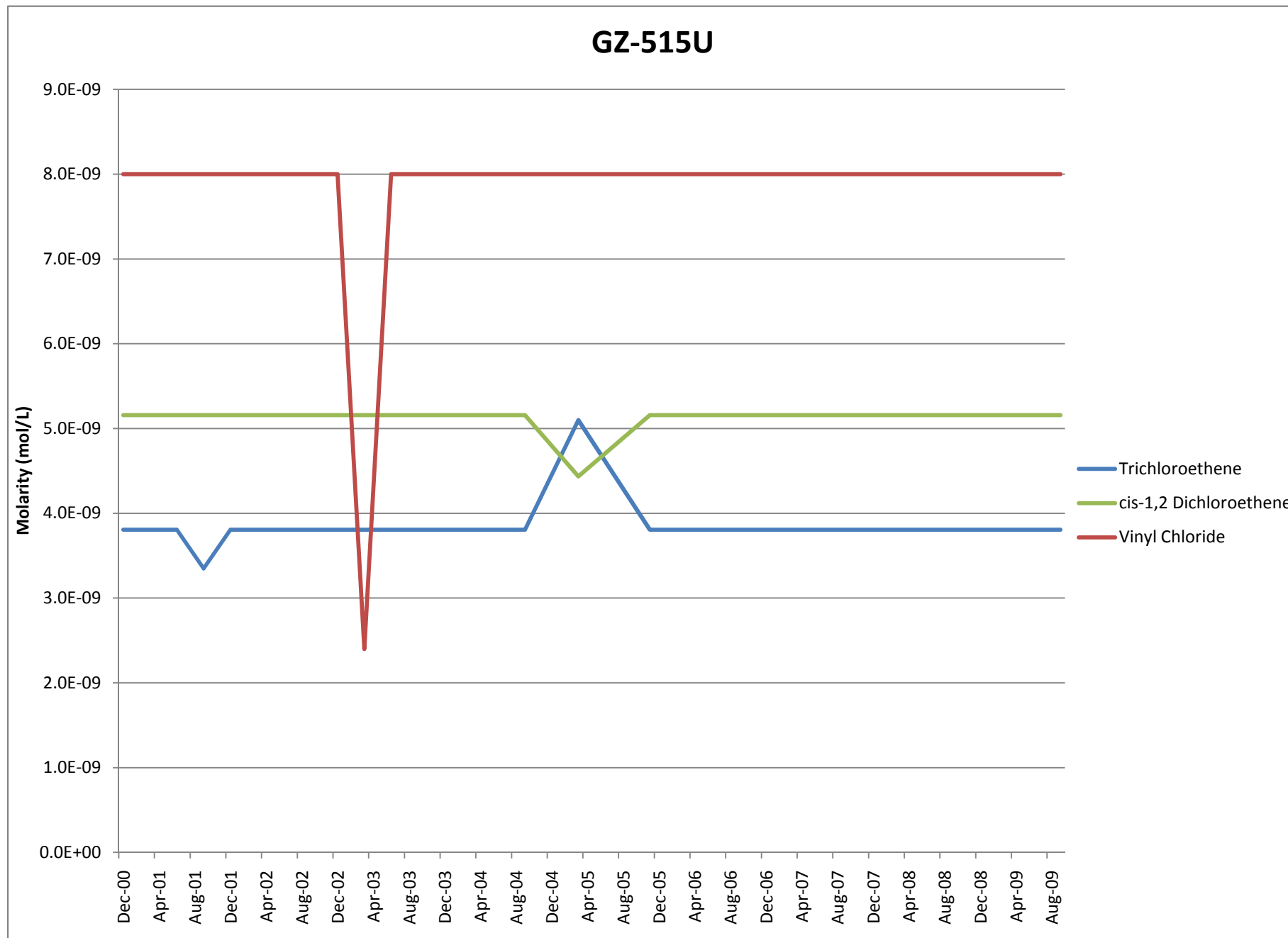


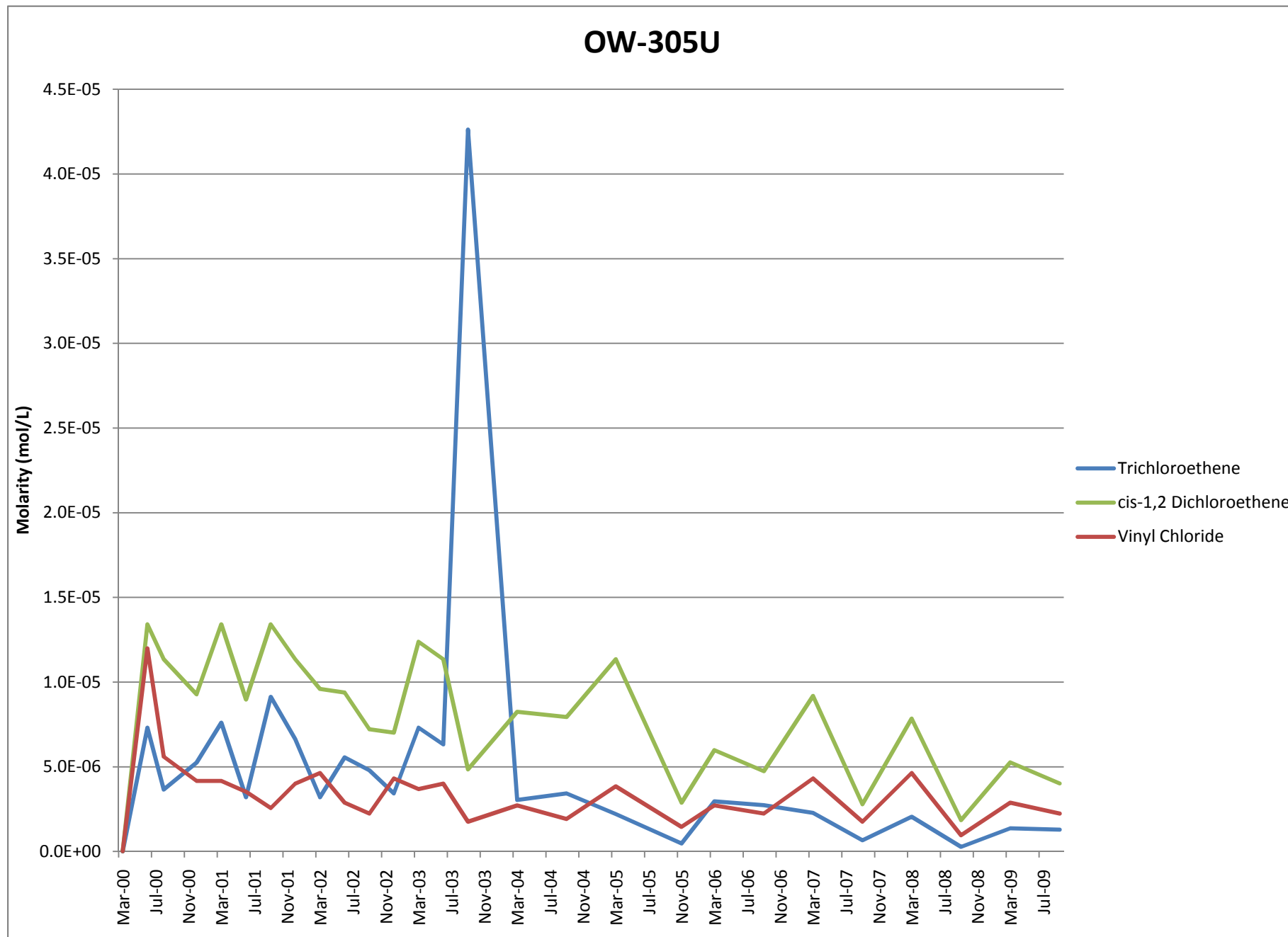




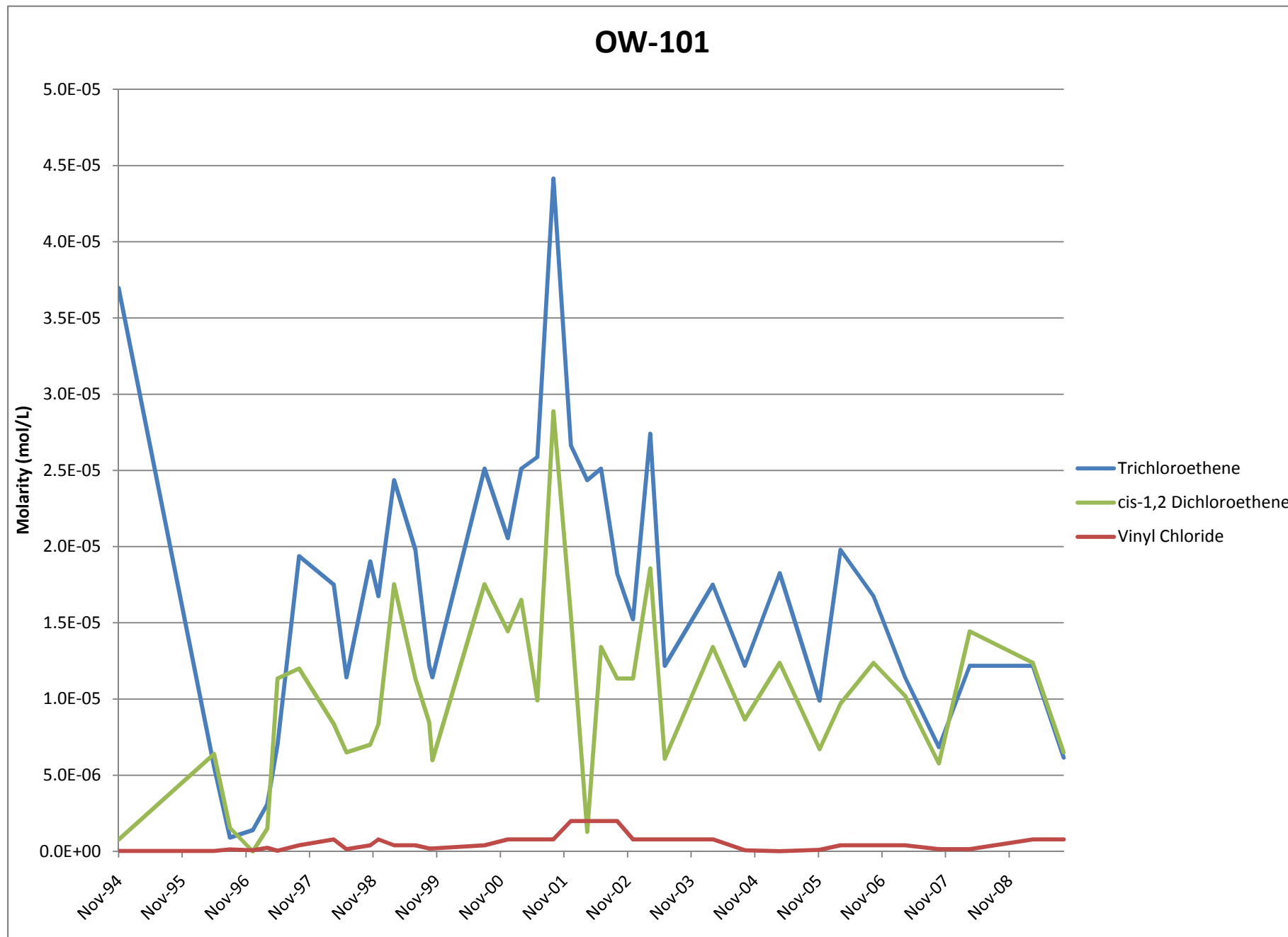


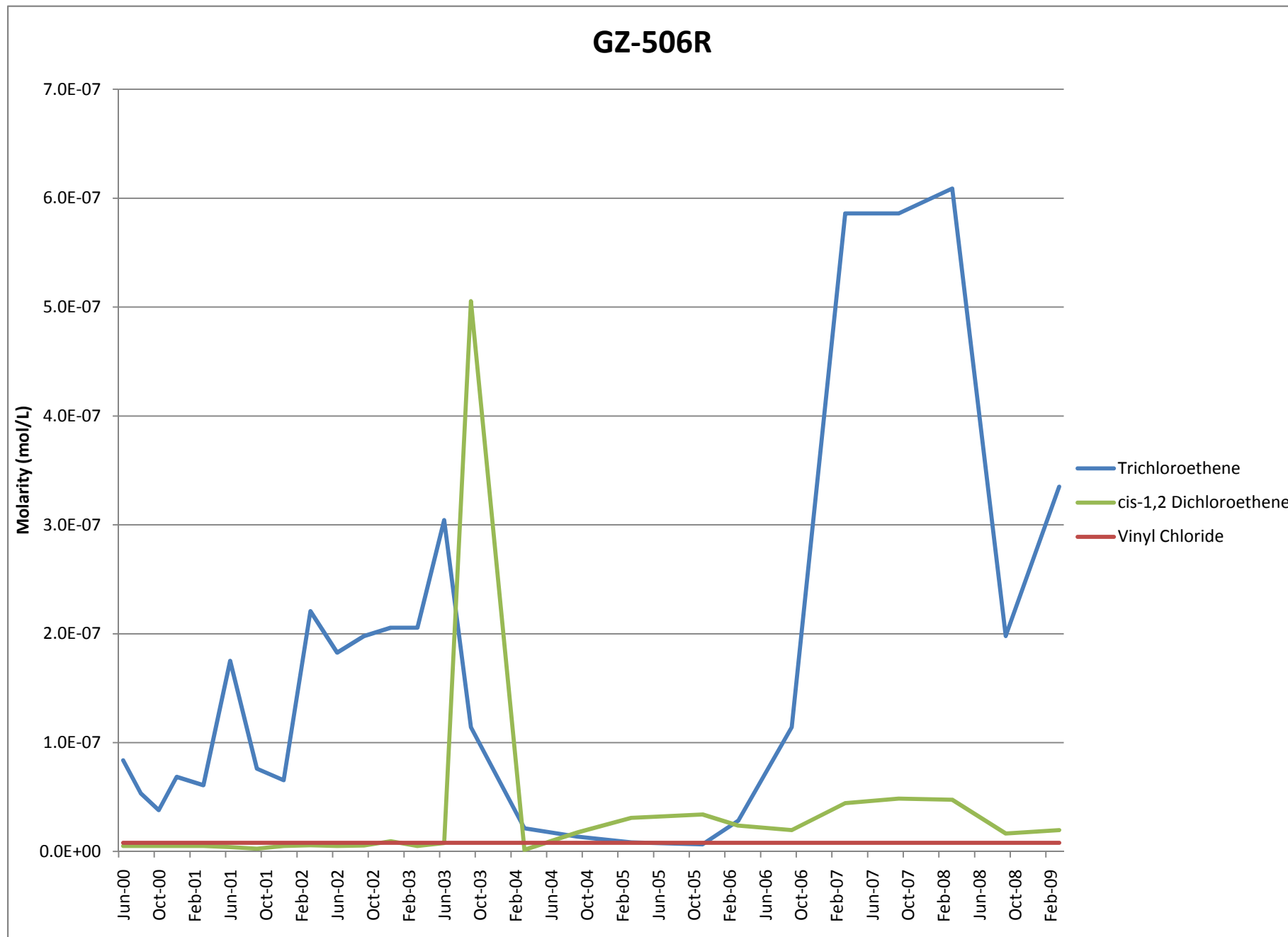


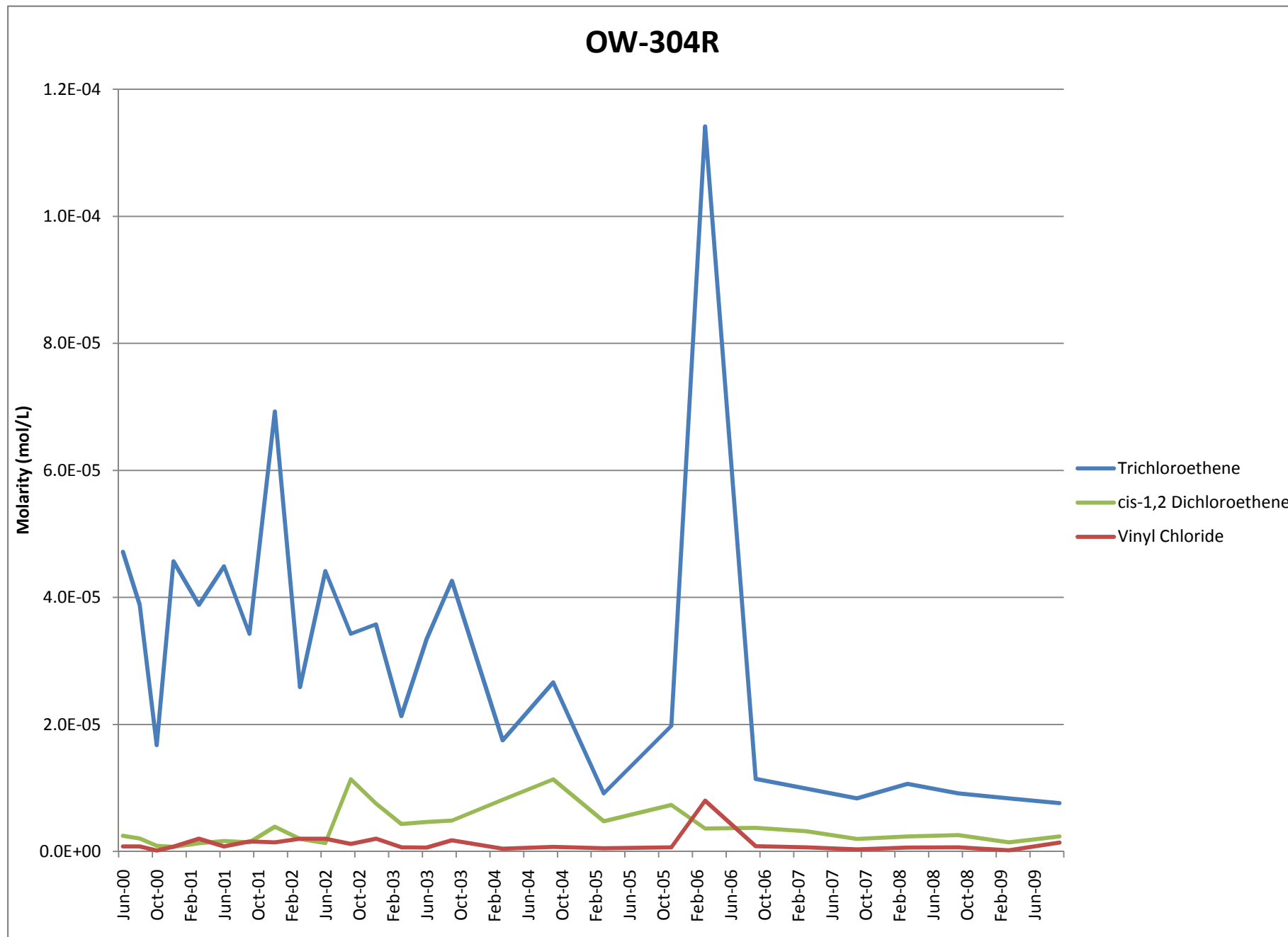


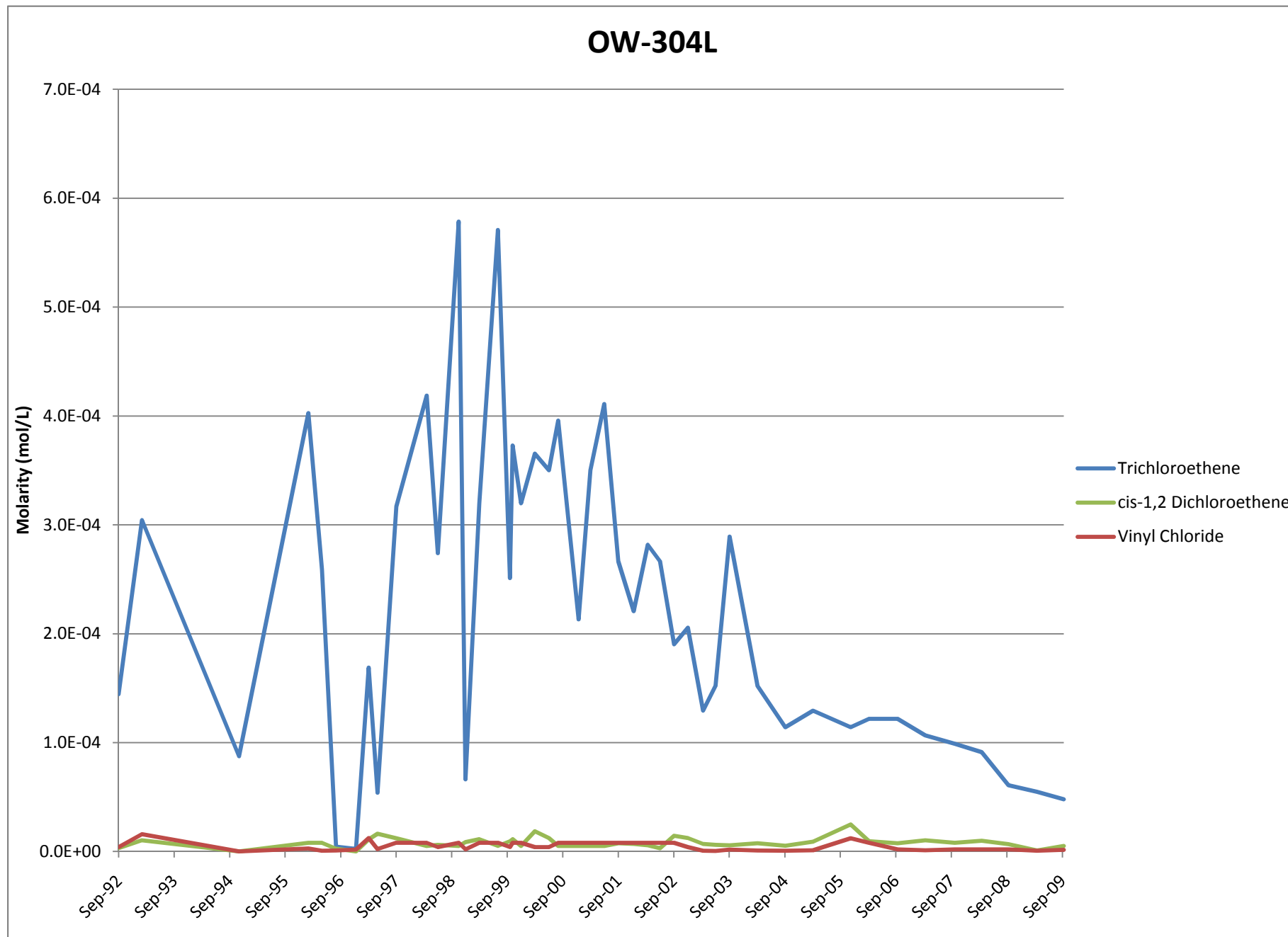


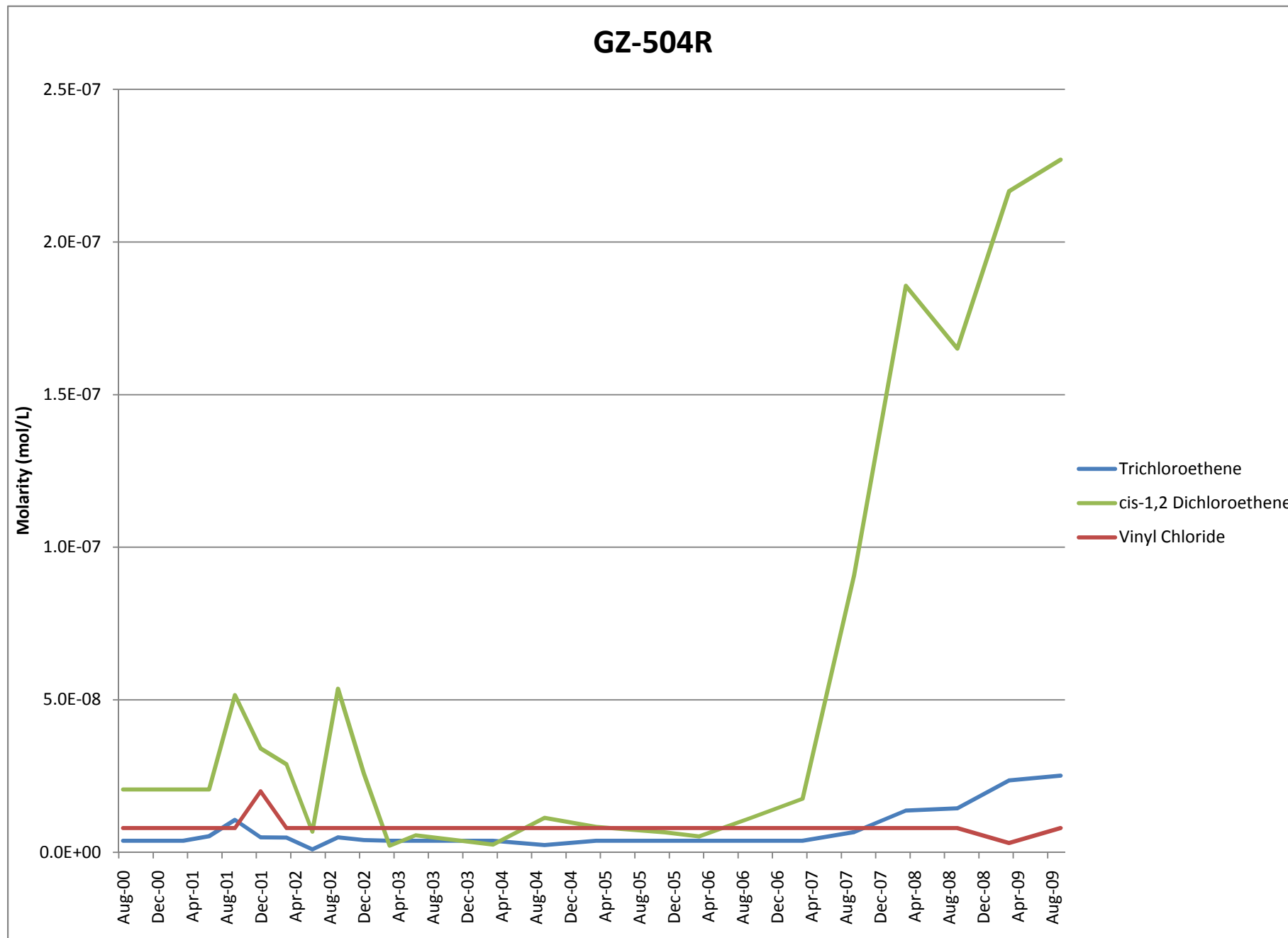


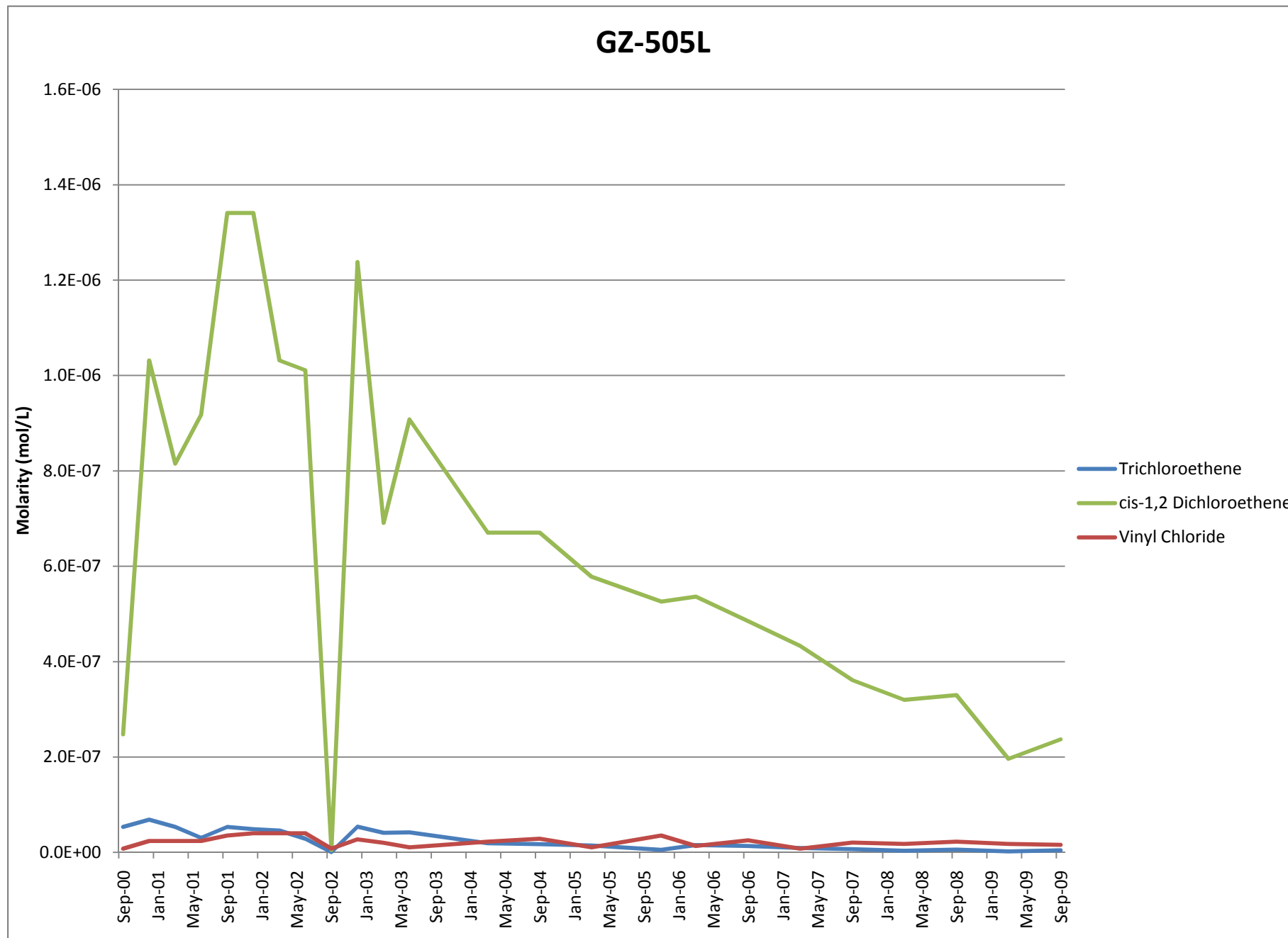


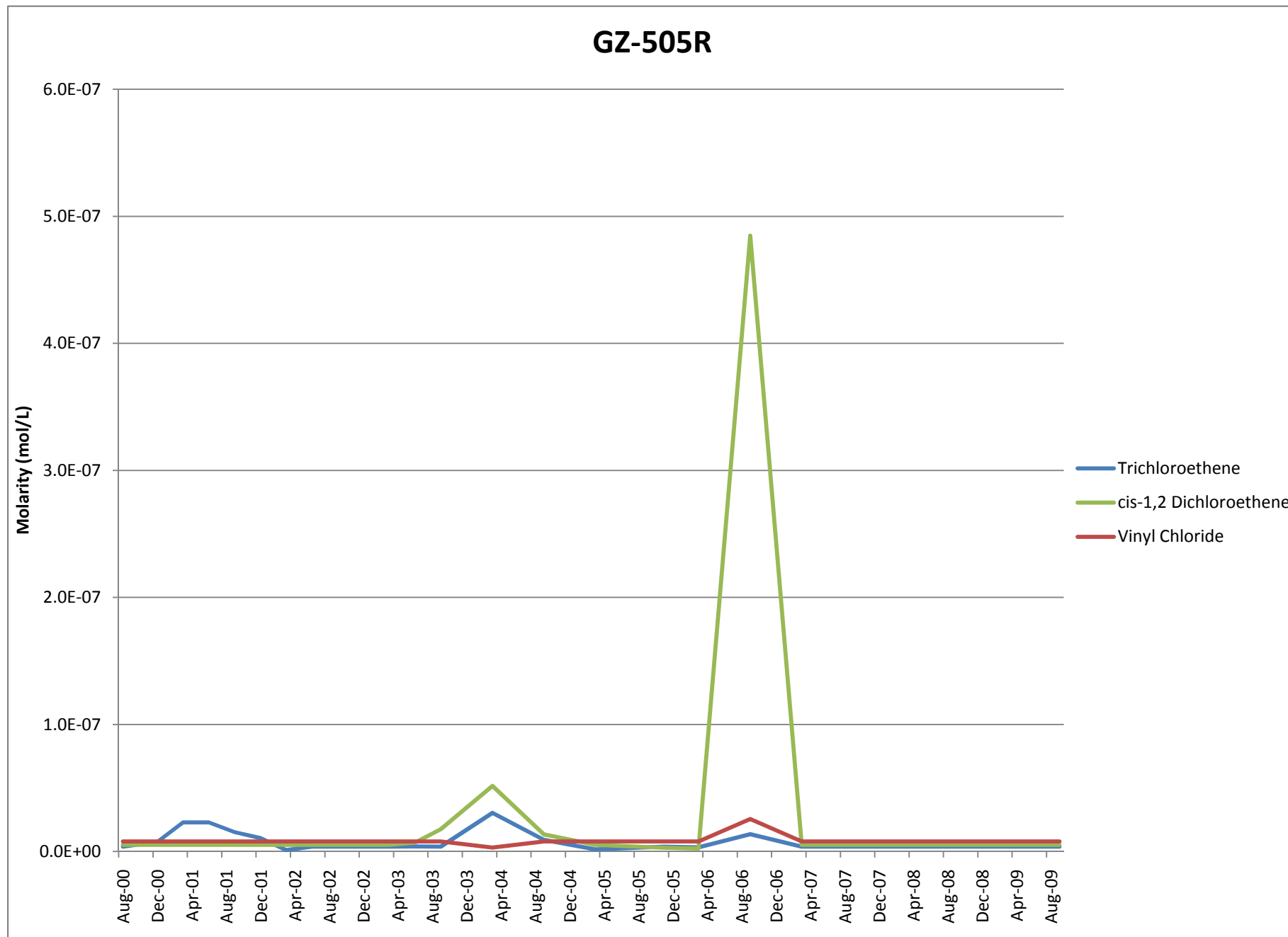


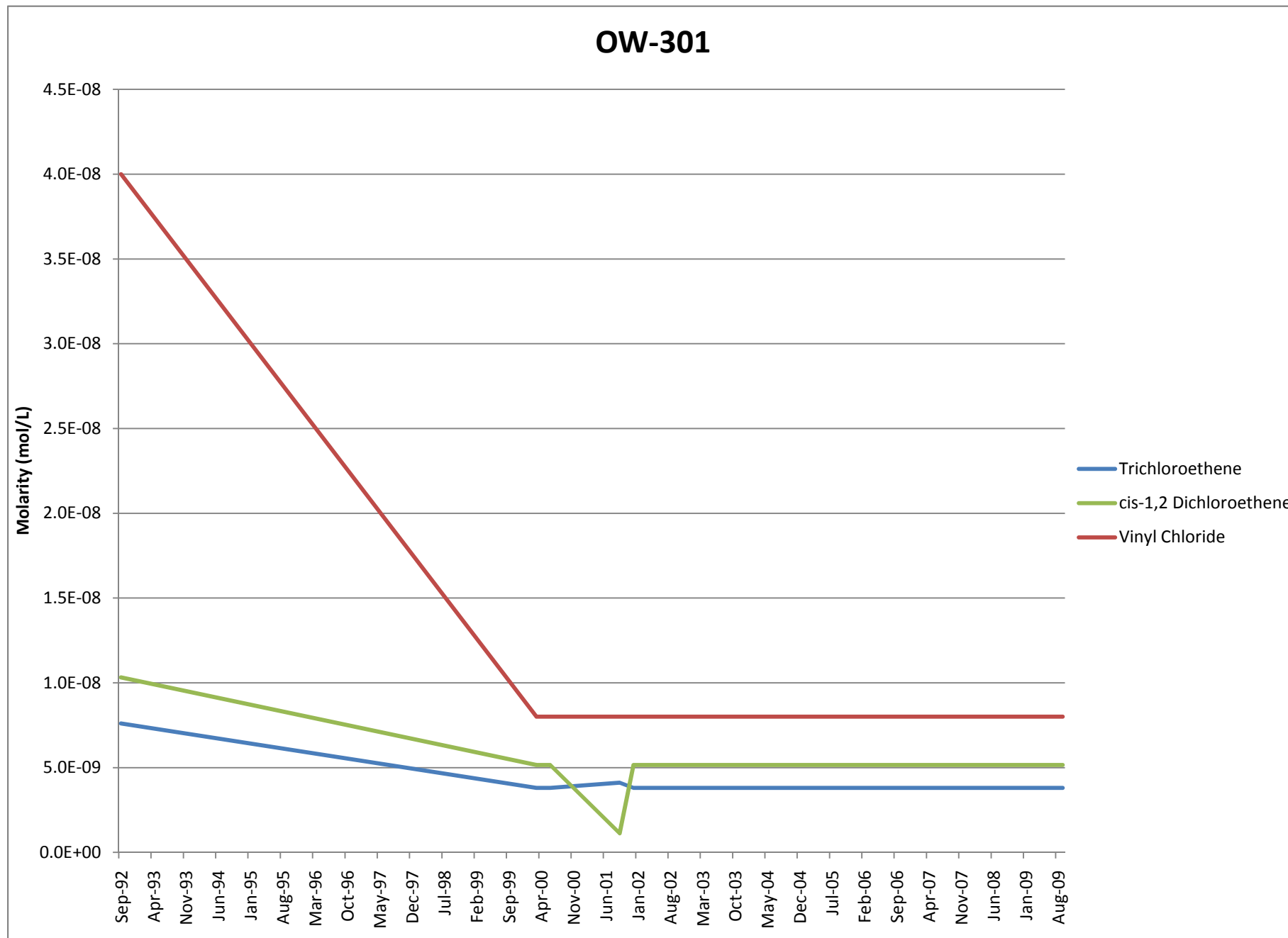




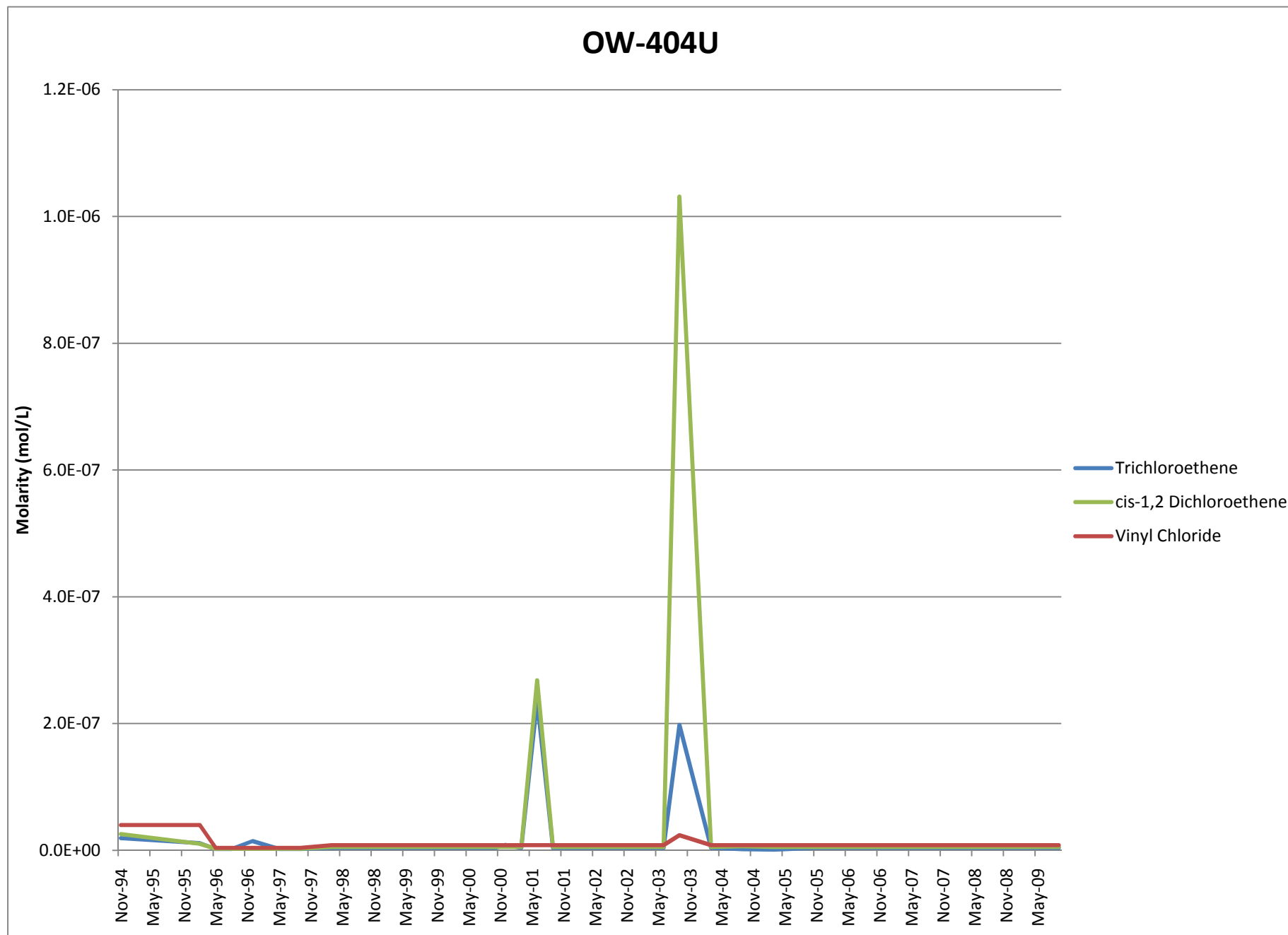












**APPENDIX D**

**QUALITY ASSURANCE PROJECT PLAN (QAPP)**

**(SUBMITTED MAY 2010)**

**APPENDIX B**  
**LIMITATIONS**

## **APPENDIX B**

### **LIMITATIONS**

1. The reported findings submitted in this report are based in part upon previous and recent data obtained from a limited number of samples from widely spaced subsurface explorations and monitoring wells. The nature and extent of variations between these explorations may not become evident until further investigation is performed. If variations or other latent conditions then appear evident, it will be necessary to re-evaluate the conclusions of this Report.
2. Water level readings have been made in the observation wells periodically and under conditions stated in the text. These data have been reviewed and interpretations have been made in the text of this Report. However, it must be noted that fluctuations in the level of the groundwater may occur due to variations in rainfall and other factors different from those prevailing at the time measurements were made.
3. Quantitative laboratory testing was performed as part of the site investigation and remediation work. Where such analyses have been conducted by an outside laboratory, GZA GeoEnvironmental, Inc. (GZA) has relied upon the data provided, and has not conducted an independent evaluation of the reliability of these data.
4. The findings contained in this Report are based in part upon various types of chemical data and are contingent upon their validity. These data have been reviewed and interpretations made in the Report. Some of these data were preliminary "screening" level data, and may have not been confirmed with quantitative analyses. Moreover, it should be noted that variations in the types and concentrations of contaminants and variations in their flow paths may occur due to seasonal water table fluctuations, past disposal practices, the passage of time, and other factors. Should additional chemical data become available in the future, these data should be reviewed by GZA, and the findings presented herein modified accordingly.
5. Chemical analyses have been performed for specific parameters during the course of this study, as detailed in the text. It must be noted that additional constituents not searched for during the current study may be present in soil and groundwater at the site.

## **APPENDIX C**

**SAFETY DATA SHEETS ABC<sup>®</sup> AND KB-1<sup>®</sup>**

**Material Safety Data Sheet**

May be used to comply with OSHA's Hazard Communication Standard, 29 CFR 1910.1200. Standard must be consulted for specific requirements.

**U.S. Department of Labor**

Occupational Safety and Health Administration  
(Non-Mandatory Form)  
Form Approved  
OMB No. 1218-0072

IDENTITY (as Used on Label and List)  
**ABC (Anaerobic BioChem)**

*Note: Blank spaces are not permitted. If any item is not applicable or no information is available, the space must be marked to indicate that.*

**Section I**

Manufacturer's name <b>REDOX TECH, LLC</b>	Emergency Telephone Number <b>919-678-0140</b>
Address (Number, Street, City, State and ZIP Code) <b>200 Quade Dr, Cary NC 27513</b>	Telephone Number for Information <b>919-678-0140</b>
	Date Prepared <b>November 2013</b>
	Signature of Preparer (optional)

**Section II—Hazardous Ingredients/Identity Information**

Hazardous Components (Specific Chemical Identity, Common Name(s))	OSHA PEL	ACGIH TLV	Other Limits Recommended	% (optional)
Mixture of :				
Tri-alcohol propane	NA	NA		(0 to 60%)
Lactic acid esters	NA	NA		(0 to 98.5%)
Phosphate buffer	NA	NA		(0 to 0.1%)
Ferrous Iron	NA	NA		(0 to 0.1%)
Fatty Acids	NA	NA		(0 to 30%)

**Section III—Physical/Chemical Characteristics**

Boiling Point	> 400 C	Specific Gravity (H <sub>2</sub> O = 1)	1.04
Vapor Pressure (mm Hg) <1		pH	6.0 to 8.0
Vapor Density (AIR = 1)		Evaporation Rate (Butyl Acetate = 1)	No information
Solubility in Water	Fully soluble and miscible		
Appearance and Odor	Light yellow to colorless liquid, slight to mild, characteristic odor		

**Section IV—Fire and Explosion Hazard Data**

Flash Point (Method Used)	Flammable Limits	LEL	UEL
Does not flash			
Extinguishing Media	Water spray, carbon dioxide, dry powder, AFFF, foam		
Special Fire Fighting Procedures			
Standard procedures for chemical fires			
Unusual Fire and Explosion Hazards			
Thermal decomposition can release irritating gases and vapors			

(Reproduce locally)

OSHA 174 Sept. 1985

<b>Section V—Reactivity Data</b>			
Stability	Unstable		Conditions to Avoid
	Stable	X	
Incompatibility ( <i>Materials to Avoid</i> ) Strong oxidants			
Hazardous Decomposition or Byproducts no known			
Hazardous Polymerization	May Occur		Conditions to Avoid
	Will Not Occur	X	
<b>Section VI—Health Hazard Data</b>			
Route(s) of Entry	Inhalation? Yes, esters only	Skin? Yes	Ingestion? Yes
Health Hazards ( <i>Acute and Chronic</i> ) Acid esters: Risk of irritation to eyes. Irritating to respiratory system. May decrease skin.			
Carcinogenicity	NTP? No	IARC Monographs? No	OSHA Regulated? No
Signs and Symptoms of Exposure Red irritated skin. May cause light-headedness when used in poorly ventilated area without proper ventilation because of ethanol vapors			
Medical Conditions			
Generally Aggravated by Exposure Persons susceptible or sensitive to eye and respiratory irritation			
Emergency and First Aid Procedures			
Inhalation: Move to fresh air; Skin: Wash skin immediately with water. Eyes: Flush with water for at least 15 minutes, consult physician; Ingestion: Drink water and consult physician.			
<b>Section VII—Precautions for Safe Handling and Use</b>			
Use safety glasses and latex or nitrile gloves. Work in well ventilated area.			
Waste Disposal Method ABC can be disposed as waste water or landfilled when in compliance with local regulations			
Precautions to Be Taken in Handling and Storing Not flammable			
Avoid long storage times, will break down to innocuous weak organic acids			
Other Precautions Wear PPE when handling and keep containers tightly closed when stored.			
<b>Section VII—Control Measures</b>			
Respiratory Protection ( <i>Specify Type</i> ) none required			
Ventilation	Local Exhaust	Standard HVAC conditions typically adequate	Special
	Mechanical ( <i>General</i> )	Floor or stand fans	Other
Protective Gloves	PVA, nitrile or latex	Eye Protection	Safety glasses with side shields
Other Protective Clothing or Equipment			
Work/Hygienic Practices Do not eat, drink or smoke while handling. Remove/wash contaminated clothing before reuse.			

## KB-1<sup>®</sup> Material Safety Data Sheet

### Section 1: Material Identification

**Trade Name:** KB-1<sup>®</sup>

**Chemical Family:** bacterial mixture

**Chemical name:** No IUC name for mixture is known to exist

**Manufacturer/Supplier:** SiREM  
130 Research Lane, Suite 2,  
Guelph, Ontario,  
Canada N1G 5G3

**For Information call:** 519-822-2265 / 1-866-251-1747 x236

**Emergency Number:** 519-822-2265

**Description:** Microbial inoculum (non-pathogenic, non-hazardous)

**Trade Name:** KB-1<sup>®</sup>

**Product Use:** Bioremediation of contaminated groundwater.

**Date Prepared:** 2 February 2005

### Section 2: Composition, Information on Ingredients

KB-1<sup>®</sup> is a microbial culture grown in an aqueous dilute mineral salt solution media containing no hazardous ingredients.

The microbial composition of KB-1<sup>®</sup> (as determined by phylogenetic analysis) is listed in Table 1. Identification of organisms was obtained by matching 16S rRNA gene sequence of organisms in KB-1<sup>®</sup> to other known organisms. The characteristics of related organisms can be used to identify potential or likely characteristics of organisms in KB-1<sup>®</sup>.

**Table 1. Genus' Identified in KB-1<sup>®</sup> Microbial Inoculum**

Genus
Dehalococcoides sp.
Geobacter sp.
Methanomethylovorans sp.

### Section 3: Hazards Identification:

A review of the available data does not indicate any known health effects related to normal use of this product.

### Section 4: First Aid Measures:

Avoid direct contact with skin and eyes. In any case of any exposure which elicits a response, a physician should be consulted immediately.

**Eye Contact:** Flush eyes with water for at least 15 minutes, occasionally lift upper and lower eyelids, if undue irritation or redness occurs seek medical attention.

**Skin Contact:** Remove contaminated clothing and wash skin thoroughly with water and antibacterial soap. Seek medical attention if irritation develops or open wounds are present.



**Ingestion:** Do not induce vomiting, drink several cups of water, seek medical attention.

**Inhalation:** Remove to fresh air. If not breathing give artificial respiration. In case of labored breathing give oxygen. Call a physician.

### **Section 5 - Fire Fighting Measures:**

Non-flammable

Flash Point: not applicable

Upper flammable limit: not applicable

Lower flammable limit: not applicable

### **Section 6 – Accidental Release Procedures**

Spilled KB-1<sup>®</sup> should be soaked up with sorbant and saturated with a 10% bleach solution (prepared by making a one in ten dilution of diluted standard bleach [normally sold at a strength of 5.25% sodium hypochlorite] to disinfect affected surfaces. Sorbant should be double bagged and disposed of as indicated in section 12. After removal of sorbant, area should be washed with 10% bleach solution to disinfect. If liquid from the culture vessel is present on the fittings, non-designated tubing or exterior of the stainless steel pressure vessel liquid should be wiped off and the area washed with 10% bleach solution.

### **Section 7 - Handling and Storage**

KB-1<sup>®</sup> is shipped in stainless steel pressure vessels and connected to injection lines and inert gas is used to pressurize the vessel to displace the contents. KB-1<sup>®</sup> should be handled with care to avoid any spillage. Vessels are shipped with 1 pound per square inch (psi) pressure; valves should not be opened until connections to appropriate lines for subsurface injection are in place.

**Storage Requirements:** Avoid exposing stainless steel pressure vessels to undue temperature extremes (i.e., temperatures less than 0°C or greater than 30°C may result in harm to the microbial cultures and damage to the vessels). All valves should be in the closed position when the vessel is not pressurized or not in use to prevent the escape of gases and to maintain anaerobic conditions in the vessel. Avoid exposure of the culture to air as the presence of oxygen will kill dechlorinating microorganisms.

### **Section 8 - Exposure Controls/Personal Protection**

#### **Personal protective equipment:**

Skin: Protective gloves (latex, vinyl or nitrile) should be worn.

Eye Protection: Wear appropriate protective eyeglasses or goggles when opening pressure vessels, valves, or when pressurizing vessels to inject contents into the subsurface.

Respiratory: No respiratory protection is required.

Engineering Controls: Good general room ventilation is expected to be adequate.

### **Section 9: Physical and Chemical Properties:**

Physical State: liquid

Odour: skunky odour

Appearance: dark grey, slightly turbid liquid under anaerobic conditions, pink if exposed to air (oxygen).

Specific gravity: not determined

Vapor pressure: not applicable

Vapor density: not applicable

Evaporation rate: not determined

Boiling point: ~100° C

Freezing point/melting point: ~ 0°C

pH: 6.5-7.5  
Solubility: fully soluble in water

### **Section 10 – Stability and Reactivity Data**

Stable and non-reactive.  
Maintain under anaerobic conditions to preserve product integrity.  
Materials to avoid: none known

### **Section 11 - Toxicological Information**

Potential for Pathogenicity:

KB-1<sup>®</sup> has tested negative (i.e., the organisms are not present) for a variety of pathogenic organisms listed in Table 2. While there is no evidence that virulent pathogenic organisms are present in KB-1<sup>®</sup>, there is potential that certain organisms in KB-1<sup>®</sup> may have the potential to act as opportunistic (mild) pathogens, particularly in individuals with open wounds and/or compromised immune systems. For this reason standard hygienic procedures such as hand washing after use should be observed.

**Table 2, Results of Human Pathogen Screening of KB-1<sup>®</sup> Dechlorinator**

<b>Organism</b>	<b>Disease(s) Caused</b>	<b>Test result</b>
<i>Salmonella</i> sp.	Typhoid fever, gastroenteritis	Not Detected
<i>Listeria monocytogenes</i>	Listerioses	Not Detected
<i>Vibrio</i> sp.,	Cholera, gastroenteritis	Not Detected
<i>Campylobacter</i> sp.,	Bacterial diarrhea	Not Detected
<i>Clostridia</i> sp.,	Food poisoning, Botulism, tetanus, gas gangrene	Not Detected
<i>Bacillus anthracis</i>	Anthrax	Not Detected
<i>Pseudomonas aeruginosa</i>	Wound infection	Not Detected
<i>Yersinia</i> sp.,	Bubonic Plague, intestinal infection	Not Detected
Yeast and Mold	Candidiasis, Yeast infection etc.	Not Detected
Fecal coliforms	Indicator organisms for many human pathogens diarrhea, urinary tract infections	Not Detected
<i>Enterococci</i>	Various opportunistic infections	Not Detected

### **Section 12. Disposal Considerations**

Material must be disinfected or sterilized prior to disposal. Consult local regulations prior to disposal.

### **Section 13 – Transport Information**

Non-hazardous, non-pathogenic microbial inoculum – Biosafety Risk Group 1.  
Chemicals, Not Otherwise Indexed (NOI), Non-hazardous  
Not subject to TDG or DOT guidelines.

**Disclaimer:**

The information provided on this MSDS sheet is based on current data and represents our opinion based on the current standard of practice as to the proper use and handling of this product under normal, reasonably foreseeable conditions.

Last revised: 2 August 2011

## **APPENDIX D**

QUALITY ASSURANCE PROJECT PLAN (QAPP) REVISION 4

**REVISED QUALITY ASSURANCE  
PROJECT PLAN REVISION 4  
HEWLETT-PACKARD  
VOLUNTARY REMEDIAL ACTIONS  
SAN GERMAN, PUERTO RICO**

**PREPARED FOR:**

Hewlett-Packard Company  
Fort Collins, Colorado

**PREPARED BY:**

GZA GeoEnvironmental, Inc.  
Norwood, Massachusetts

April 2015  
Revised October 2015  
File No. 01.0024065.16

## TABLE OF CONTENTS

	<u>PAGE</u>
QAPP Worksheet #1: Title and Approval Page.....	1
QAPP Worksheet #2: QAPP Identifying Information.....	3
QAPP Worksheet #3: Distribution List .....	7
QAPP Worksheet #4-1: Project Personnel Sign-Off Sheet .....	8
QAPP Worksheet #4-2: Project Personnel Sign-Off Sheet .....	9
QAPP Worksheet #5: Project Organizational Chart.....	10
QAPP Worksheet #6: Communication Pathways.....	10
QAPP Worksheet #7: Personnel Responsibilities and Qualifications Table .....	12
QAPP Worksheet #8: Special Personnel Training Requirements Table .....	13
QAPP Worksheet #9: Project Scoping Session Participants Sheet .....	14
QAPP Worksheet #10: Problem Definition.....	15
QAPP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements.....	16
QAPP Worksheet #12: Measurement Performance Criteria Table .....	17
QAPP Worksheet #13: Secondary Data Criteria and Limitations Table.....	18
QAPP Worksheet #14: Summary of Project Tasks .....	19
QAPP Worksheet #15: Reference Limits and Evaluation Table .....	21
QAPP Worksheet #16: Project Schedule/Timeline Table .....	22
QAPP Worksheet #17: Sampling Design and Rationale .....	23
QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements Table .....	24
QAPP Worksheet #19: Analytical SOP Requirements Table.....	26
QAPP Worksheet #20: Field Quality Control Sample Summary Table.....	27
QAPP Worksheet #21: Project Sampling SOP References Table .....	28
QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table .....	29
QAPP Worksheet #23: Analytical SOP References Table .....	31
QAPP Worksheet #24: Analytical Instrument Calibration Table.....	32

## TABLE OF CONTENTS

	<u>PAGE</u>
QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table .....	33
QAPP Worksheet #26: Sample Handling System .....	34
QAPP Worksheet #27: Sample Custody Requirements .....	35
QAPP Worksheet #28: QC Samples Table.....	36
QAPP Worksheet #29: Project Documents and Records Table .....	38
QAPP Worksheet #30: Analytical Services Table.....	39
QAPP Worksheet #31: Planned Project Assessments Table .....	40
QAPP Worksheet #32: Assessment Findings and Corrective Action Responses.....	41
QAPP Worksheet #33: QA Management Reports Table.....	42
QAPP Worksheet #34: Verification (Step I) Process Table .....	43
QAPP Worksheet #35: Validation (Steps IIa and IIb) Process Table .....	45
QAPP Worksheet #36: Validation (Steps IIa and IIb) Summary Table .....	46
QAPP Worksheet #37: Usability Assessment .....	47

## FIGURES

FIGURE 1     SITE PLAN

## APPENDICES

APPENDIX A	LABORATORY STANDARD OPERATING PROCEDURES (TESTAMERICA)
APPENDIX B	EXAMPLE DOCUMENTS
APPENDIX C	GENERAL LABORATORY QUALITY CONTROL GUIDELINES
APPENDIX D	SAMPLING STANDARD OPERATING PROCEDURES (GZA)
APPENDIX E	EQUIPMENT USER MANUALS

## QAPP Worksheet #1: Title and Approval Page

**Site Name/Project Name:** Hewlett-Packard Voluntary Remedial Actions  
**Site Location:** San German, Puerto Rico

Quality Assurance Project Plan – Revision 4, Hewlett-Packard Voluntary Remedial Actions  
Document Title

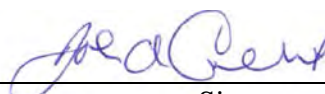
Hewlett-Packard Company  
Lead Organization

James Roehrig - GZA GeoEnvironmental, Inc  
Preparer's Name and Organizational Affiliation

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14/10/2015  
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: \_\_\_\_\_

  
Signature

John A. Colbert, PE - GZA GeoEnvironmental, Inc.      14/10/2015  
Printed Name/Organization/Date

Investigative Organization's Project QA Officer: \_\_\_\_\_

  
Signature

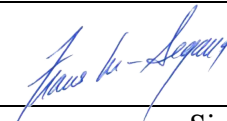
Chunhua Liu, Sc.D., GZA GeoEnvironmental, Inc.      14/10/2015  
Printed Name/Organization/Date

Lead Organization's Project Manager: \_\_\_\_\_

  
Signature

Paul E. Paschke, Hewlett-Packard Company      14/10/2015  
Printed Name/Organization/Date

PREQB QA/QC Specialist Manager: \_\_\_\_\_

  
Signature

Mrs. Frances M. Segarra Roman  
Printed Name/Organization/Date

Approval Signatures: \_\_\_\_\_

Signature

Printed Name/Title/Date



Puerto Rico Environmental Quality Board

Approval Authority

Other Approval Signatures: \_\_\_\_\_

Signature

Jesse Avilés, USEPA Region 2

Printed Name/Title/Date

## QAPP Worksheet #2: QAPP Identifying Information

**Site Name/Project Name:** Hewlett-Packard Voluntary Remedial Actions

**Site Location:** San German, Puerto Rico

**Site Number/Code:** N/A

**Operable Unit:** N/A

**Contractor Name:** GZA GeoEnvironmental, Inc.

**Contractor Number:** PRD991291857 (EPA ID)

**Contract Title:** N/A

**Work Assignment Number:** N/A

1. Identify guidance used to prepare QAPP:

Uniform Federal Policy for Quality Assurance Project Plans

2. Identify regulatory program: Voluntary remedial action

3. Identify approval entity: Puerto Rico Environmental Quality Board (EQB)

4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)

5. List dates of scoping sessions that were held: December 2, 2014

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
<u>Quality Assurance Project Plan – Revision 1</u>	<u>8/25/2000</u>
<u>Quality Assurance Project Plan – Revision 2</u>	<u>11/3/2006</u>
<u>Quality Assurance Project Plan – Revision 3</u>	<u>11/1/2010</u>

7. List organizational partners (stakeholders) and connection with lead organization:

Lead organization, Hewlett-Packard Company of Palo Alto, California, is responsible for the Voluntary Remedial Action. Hewlett-Packard has retained GZA GeoEnvironmental, Inc. of Norwood, Massachusetts as their lead environmental consultant. The land on which the remedial actions are being conducted is owned by Puerto Rico Industrial Development Company (PRIDCO) of San Juan, Puerto Rico.

8. List data users: Hewlett-Packard Company; Puerto Rico Environmental Quality Board; US Environmental Protection Agency – Region 2; GZA GeoEnvironmental, Inc.

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below: Not applicable

**QAPP Worksheet #2**  
**QAPP Identifying Information**  
**(continued)**

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
<b>Project Management and Objectives</b>		
2.1 Title and Approval Page	- Title and Approval Page	1-2
2.2 Document Format and Table of Contents	- Table of Contents	i
2.2.1 Document Control Format	- QAPP Identifying Information	3-6
2.2.2 Document Control Numbering System		
2.2.3 Table of Contents		
2.2.4 QAPP Identifying Information		
2.3 Distribution List and Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	7 8-10
2.3.1 Distribution List	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	11 12 13 14
2.3.2 Project Personnel Sign-Off Sheet		
2.4 Project Organization		
2.4.1 Project Organizational Chart		
2.4.2 Communication Pathways		
2.4.3 Personnel Responsibilities and Qualifications	- Special Personnel Training Requirements Table	14
2.4.4 Special Training Requirements and Certification		
2.5 Project Planning/Problem Definition	- Project Planning Session Documentation (including Data Needs tables)	N/A
2.5.1 Project Planning (Scoping)	- Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	15
2.5.2 Problem Definition, Site History, and Background		16
		Figure 1
2.6 Project Quality Objectives and Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	17 18
2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process		
2.6.2 Measurement Performance Criteria		
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	19 19
2.8 Project Overview and Schedule	- Summary of Project Tasks	20-21
2.8.1 Project Overview	- Reference Limits and Evaluation Table	22
2.8.2 Project Schedule	- Project Schedule/Timeline Table	23

**QAPP Worksheet #2**  
**QAPP Identifying Information**  
**(continued)**

<b>Measurement/Data Acquisition</b>		
3.1 Sampling Tasks	- Sampling Design and Rationale	24
3.1.1 Sampling Process Design and Rationale		
3.1.2 Sampling Procedures and Requirements	- Sample Location Map	Figure 1
3.1.2.1 Sampling Collection Procedures	- Sampling Locations and Methods/ SOP Requirements Table	25-26
3.1.2.2 Sample Containers, Volume, and Preservation		
3.1.2.3 Equipment/Sample Containers	- Analytical Methods/SOP Requirements Table	27
Cleaning and Decontamination Procedures		
3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures	- Field Quality Control Sample Summary Table	28
3.1.2.5 Supply Inspection and Acceptance Procedures	- Sampling SOPs - Project Sampling SOP	Appendix D 29
3.1.2.6 Field Documentation Procedures	References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	30-31
3.2 Analytical Tasks	- Analytical SOPs	Appendix A
3.2.1 Analytical SOPs	- Analytical SOP References Table	32
3.2.2 Analytical Instrument Calibration Procedures	- Analytical Instrument Calibration Table	33
3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures	- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	34
3.2.4 Analytical Supply Inspection and Acceptance Procedures		
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures	- Sample Collection Documentation Handling, Tracking, and Custody SOPs	Appendix D
3.3.1 Sample Collection Documentation		
3.3.2 Sample Handling and Tracking System	- Sample Container Identification	36
3.3.3 Sample Custody	- Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	35 Appendix B
3.4 Quality Control Samples	- QC Samples Table	37-38
3.4.1 Sampling Quality Control Samples	- Screening/Confirmatory Analysis Decision Tree	N/A
3.4.2 Analytical Quality Control Samples		
3.5 Data Management Tasks	- Project Documents and Records Table	39
3.5.1 Project Documentation and Records		
3.5.2 Data Package Deliverables	- Analytical Services Table	40
3.5.3 Data Reporting Formats	- General Guidelines for Quality Control	Appendix C
3.5.4 Data Handling and Management		
3.5.5 Data Tracking and Control		

**QAPP Worksheet #2**  
**QAPP Identifying Information**  
**(continued)**

<b>Assessment/Oversight</b>		
4.1 Assessments and Response Actions	- Assessments and Response Actions	Appendix A (lab)
4.1.1 Planned Assessments	- Planned Project Assessments Table	42
4.1.2 Assessment Findings and Corrective Action Responses	- Audit Checklists - Assessment Findings and Corrective Action Responses Table	Appendix B 41
4.2 QA Management Reports	- QA Management Reports Table	43
4.3 Final Project Report		
<b>Data Review</b>		
5.1 Overview		
5.2 Data Review Steps	- Verification (Step I) Process Table	44-45
5.2.1 Step I: Verification		
5.2.2 Step II: Validation	- Validation (Steps IIa and IIb) Process Table	46
5.2.2.1 Step IIa Validation Activities		
5.2.2.2 Step IIb Validation Activities	- Validation (Steps IIa and IIb) Summary Table	47
5.2.3 Step III: Usability Assessment		
5.2.3.1 Data Limitations and Actions from Usability Assessment	- Usability Assessment	48-50
5.2.3.2 Activities		
5.3 Streamlining Data Review		
5.3.1 Data Review Steps To Be Streamlined		
5.3.2 Criteria for Streamlining Data Review		
5.3.3 Amounts and Types of Data Appropriate for Streamlining		

### QAPP Worksheet #3: Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address
Paul Paschke	Environmental Program Manager	Hewlett-Packard Company	970-898-0573	970-778-4192	paul.paschke@hp.com
Charles Lindberg	Senior Principal	GZA	781-278-3830	781-278-5701	charles.lindberg@gza.com
John Colbert	Senior Project Manager	GZA	781-278-5892	781-278-5701	john.colbert@gza.com
Jess Hornsby	Project Manager	TestAmerica - Tallahassee	813-885-7427	850-878-9504	jess.hornsby@testamericainc.com
Daliz M. Estades Santaliz	Puerto Rico Certified Chemist	Daliz M. Estades Santaliz	787-691-0250		destades@gmail.com
Gloria M. Toro Agrait	Project Manager	EQB	787-767-8181 ext. 3586	787-767-8118	gloriatoro@jca.gobierno.pr
Frances M. Segarra Roman	QA/QC Specialist Manager	EQB	787-767-8181 ext. 3575	787-767-8118	francessegarra@jca.pr.gov
Jesse Avilés	Project Manager	EPA Region 2	787-977-5882	787-289-7104	aviles.jesse@epa.gov

### QAPP Worksheet #4-1: Project Personnel Sign-Off Sheet

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

**Organization:** GZA GeoEnvironmental, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read

### QAPP Worksheet #4-2: Project Personnel Sign-Off Sheet

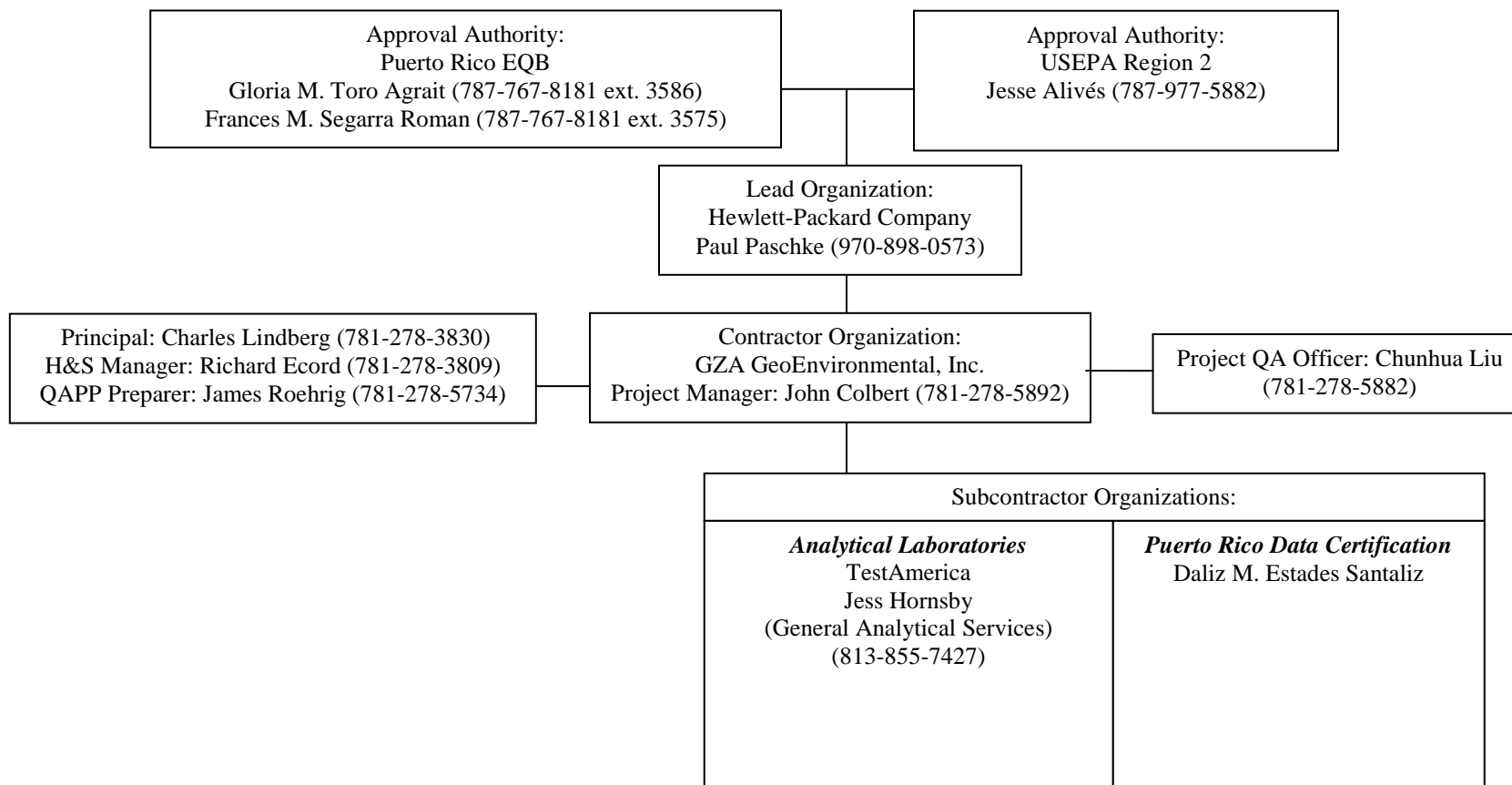
Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

**Organization:** TestAmerica

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read



## QAPP Worksheet #5: Project Organizational Chart



### QAPP Worksheet #6: Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Point of contact with EPA and EQB	Contractor Project Manager	John Colbert	781-278-5892	All required materials and information about the project will be forwarded to Jesse Avilés and Gloria Toro Agrait by John Colbert for Paul Paschke.
Manage all project phases	Contractor Project	John Colbert	781-278-5892	John Colbert will be GZA's liaison to Paul Paschke.
Manage field-related issues	Contractor Project Manager	John Colbert	781-278-5892	Notify John Colbert of field problems by phone or email as soon as possible or no later than COB the next day.
QAPP changes in the field	Sampling Team Leader	James Roehrig	781-278-5734	Notify John Colbert by phone and email of changes to the QAPP made in the field and reasons within 2 business days.
Field Reports	Sampling Team Leader	James Roehrig	781-278-5734	James Roehrig will complete field reports within 30 days of field activities.
Reporting Lab Data Quality Issues	Laboratory Project Manager	Jess Hornsby	813-885-7427	All QA/QC issues with project field samples will be reported by Jess Hornsby to John Colbert by email or phone within 2 business days.
Analytical Corrective Action	Laboratory Project Manager	Jess Hornsby	813-885-7427	The need for corrective action for analytical issues will be determined by Jess Hornsby.
Field and Analytical Corrective Action	Contractor Project Manager	John Colbert	781-278-5892	The need for corrective action for field and analytical issues will be determined by John Colbert.
Release of Analytical Data	Contractor Principal	Charles Lindberg	781-278-3830	No analytical data can be released until validation is completed and Charles Lindberg has approved the release.
QAPP Amendments	EPA Project Manager EQB Project Manager EQB QA/QC Specialist Manager	Jesse Avilés Gloria Toro Agrait Frances M. Segarra Roman	787-977-5882 787-767-8181 ext. 3586 787-767-8181 ext. 3575	Any major changes to the QAPP must be approved by Jesse Avilés, Gloria Toro Agrait, and Frances Segarra Roman before the changes can be implemented. <sup>1</sup>

<sup>1</sup> Major changes to the QAPP that would require pre-approval include using alternative SOP's from what is in this document.

**QAPP Worksheet #7: Personnel Responsibilities and Qualifications Table**

<b>Name</b>	<b>Title</b>	<b>Organizational Affiliation</b>	<b>Responsibilities</b>	<b>Education and Experience Qualifications</b>
Paul Paschke	Environmental Program Manager	HP	Oversees project	BS, Geology, 31 yr exp
Charles Lindberg	Senior Principal	GZA	Oversees project	BS, Civil Engineering, Licensed Site Professional, 36 yr exp
John Colbert	Senior Project Manager	GZA	Manages project – coordinates between lead agency and subcontractors and regulatory agencies	BS Engineering, Professional Engineer, 20 yr exp
Chunhua Liu	QA Officer/Risk Assessor	GZA	QA Oversight and performs risk assessment	ScD, Environmental Chemistry, 15 yr exp
James Roehrig	QAPP Preparer/Project Engineer	GZA	Prepares QAPP, supervises field sampling and coordinates all field activities, performs site inspections/audits	BS, Environmental Engineering, Professional Engineer, 5 yr exp,
Richard Ecord	H&S Manager	GZA	Oversees H&S for field activities performed by GZA	BS, Chemistry, Certified Industrial Hygienist, Certified Safety Professional, 23 yr exp
Jess Hornsby	Project Manager	TestAmerica	Manages generation of analytical data	BA, 13 yr exp
Jess Hornsby	Lab QAO	TestAmerica	Performs lab QA oversight	BS, Biology, 20 yr exp
Daliz M. Estades Santaliz	PR Certified Chemist	Daliz M. Estades Santaliz	Certifies analytical data performed by lab outside of Puerto Rico	MS, Environmental Management, 21 years exp

**QAPP Worksheet #8: Special Personnel Training Requirements Table**

<b>Project Function</b>	<b>Specialized Training – Title or Description of Course</b>	<b>Training Provider</b>	<b>Training Date</b>	<b>Personnel/Groups Receiving Training</b>	<b>Personnel Titles/ Organizational Affiliation</b>	<b>Location of Training Records/Certificates</b>
Sample Collection and other Field Activities	OSHA 40-hour training			All Field Staff		Subcontractor maintains training records
Laboratory Certification	Puerto Rico Data Certification			Daliz M. Estades Santaliz	Puerto Rico Certified Chemist	Subcontractor maintains training records

## QAPP Worksheet #9: Project Scoping Session Participants Sheet

<b>Project Name: Voluntary Remedial Actions</b> <b>Projected Date(s) of Sampling: Ongoing</b> <b>Project Manager: Paul Paschke (HP) &amp; John Colbert (GZA)</b>				<b>Site Name: Hewlett-Packard Voluntary Remedial Actions</b> <b>Site Location: San German, Puerto Rico</b>	
<b>Date of Session: December 2, 2014</b> <b>Scoping Session Purpose: Revisions to QAPP</b>					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Paul Paschke	Environmental Program Manager	HP	970-898-0573	paul.paschke@hp.com	Lead Organization – Project Manager
Charles Lindberg	Senior Principal	GZA	781-278-3830	charles.lindberg@gza.com	Contractor – Principal
John Colbert	Senior Project Manager	GZA	781-278-5892	john.colbert@gza.com	Contractor – Project Manager
James Roehrig	Assistant Project Manager	GZA	781-278-5833	james.roehrig@gza.com	QAPP Preparer

Comments/Decisions: A formal scoping session with all of the stakeholders was deemed unnecessary as this is the fourth revision to the QAPP.

Action Items: Obtain new/updated SOPs; Update PQOs; Update schedule/timeline; Update usability assessment

Consensus Decisions: QAPP to be finalized by April 2015

## QAPP Worksheet #10: Problem Definition

### Problem Definition

Hewlett-Packard Company (HP) voluntarily operated a Groundwater Containment and Treatment System (GWCTS) at the former Digital Equipment Corporation (Digital) facility based on groundwater contamination in exceedance of Puerto Rico Water Quality Standards (PRWQS); however, HP deactivated this system to evaluate the effectiveness and applicability of intrinsic biodegradation with approval from the Puerto Rico Environmental Quality Board (PREQB) on November 1, 2010. Historical groundwater samples collected indicate that dechlorination is occurring naturally at the Site. Groundwater contamination, primarily including trichloroethylene (TCE) and its breakdown or daughter component cis-1,2-dichloroethylene (1,2-DCE), at the Site is a result of historical use of the facility by Digital. Compaq Computer Corporation had acquired Digital in 1998 and HP and Compaq Computer Corporation merged in 2002.

### Project Description

The objective of the Intrinsic Biodegradation (IB) study is to evaluate whether, in the absence of any additional remedial technologies, intrinsic biodegradation of the residual chlorinated volatile organic compound (cVOCs) in groundwater can continue to reduce dissolved concentrations of the cVOCs while posing no additional risk to human or environmental receptors. The principal objectives of groundwater monitoring for the IB study are three-fold: (i) to evaluate whether dissolved concentrations of the cVOCs at the Site continue to decline; (ii) to monitor the relative concentrations of TCE and its daughter compounds, as well as other known biologically sensitive parameters, in order to confirm that biodegradation is occurring and to confirm the respective degradation pathway; and (iii) to monitor conditions at perimeter wells to evaluate the potential for off-Site migration of impacted groundwater. Additionally, in an effort to evaluate the efficacy of Enhanced Reductive Dechlorination (ERD) as a cVOC management of migration measure at the Site, HP is proposing an ERD pilot test targeting the areas in the vicinity of monitoring wells OW-307 and OW-101.

### Project Decision Conditions

The contaminant concentrations may take several years to stabilize on Site following the ERD pilot test. Additionally, it may take several rounds of subsequent data collection to gain clarity and certainty that steady state has been established, and as a result the verification of temporal and/or spatial concentration trends will be a protracted process. However, if an interim unfavorable condition is observed and confirmed, corrective actions will be implemented. Unfavorable conditions that would be of particular concern include confirmed trends demonstrating significantly increasing concentrations of TCE in groundwater, reversal of daughter-product dominant concentrations, absence of supporting data regarding intrinsic biodegradation activity from secondary lines of evidence and/or evidence of plume advancement. If such conditions are suspected and confirmed, additional remedial response actions will be considered, including additional ERD injections or *in-situ* chemical oxidation for localized areas and/or other remedial approaches that are deemed appropriate for the given condition. Similarly, if groundwater contouring indicates that flow patterns change significantly, additional monitoring wells may be installed downgradient of Site in the direction of groundwater flow. Since implementation of the IB study, seven additional wells have been installed on- and off-Site.

## QAPP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements

**Who will use the data?**

Data will be used by GZA to generate status reports. Data will be presented to regulatory agencies (USEPA and EQB) in these status reports.

**What will the data be used for?**

GZA will use the data to evaluate the efficacy of the remedial actions, including intrinsic biodegradation.

**What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)**

Due to Site historical use, VOCs analyses (including cVOCs) are needed. To evaluate the efficacy of intrinsic biodegradation of the Site constituents, analyses of biodegradation parameters (dissolved iron, total organic carbon, pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), methane, ethane, and ethane) are needed. Three of those parameters (pH, ORP, and DO) will be measured in the field. Field sampling and laboratory methods will follow appropriate SOPs.

**How “good” do the data need to be in order to support the environmental decision?**

Samples must support evaluations of risk and efficacy of the remedial actions. The VOC data collected will be evaluated by data usability assessments.

**How much data are needed? (number of samples for each analytical group, matrix, and concentration)**

Number of samples must be sufficient for evaluations of extent of groundwater impact, risk and efficacy of the remedial actions.

**Where, when, and how should the data be collected/generated?**

Data will be collected at the Site on a schedule to be determined by Site conditions. Sampling method will follow the appropriate SOP.

**Who will collect and generate the data?**

Data collection will be performed under the direction of GZA. Generation of the data will be performed by TestAmerica.

**How will the data be reported?**

The data will be reported in status reports.

**How will the data be archived?**

The data will be archived by GZA on backed-up servers for 5 years.

**QAPP Worksheet #12: Measurement Performance Criteria Table**

<b>Matrix</b>	Groundwater				
<b>Analytical Group</b>	VOCs				
<b>Concentration Level</b>	High/Low				
<b>Sampling Procedure</b>	<b>Analytical Method/SOP</b>	<b>Data Quality Indicators (DQIs)</b>	<b>Measurement Performance Criteria <sup>1</sup></b>	<b>QC Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&amp;A)</b>
3.1.2	8260B/1	Precision – Lab, Field	RPD < 20%	LCS/LCSD; MS/MSD	LCS/LCSD – A MS/MSD – S&A
		Accuracy/Bias - Lab	70% < %R < 130%	LCS/LCSD; MS/MSD	LCS/LCSD – A MS/MSD – S&A
		Accuracy/Bias - Contamination	No target compounds above laboratory reporting limit	Method blank, trip blank	Method blank – A Trip blank – S&A
		Precision - Field	RPD < 20%	Field duplicate; MS/MSD	S&A
		Completeness	> 90% usable laboratory analysis	Data completeness check	A
		Sensitivity	50-150% recovery at method detection limit (MDL) (guidance)	MDL study	A
		Accuracy/Bias	≤ 20%RSD; RRF > 0.050	Initial Calibration	A
		Accuracy/Bias	≤ 20%D; RRF > 0.050	Opening & Closing Continuing Calibration Verification	A
		Accuracy/Bias	80% < %R < 120%	Surrogates	A
		Accuracy/Bias	<30 second change in retention time compared with last initial calibration; <factor of 2 (-50% to +100%) change in internal standard area compared with initial calibration	Internal Standard	A

<sup>1</sup> The Measurement Performance Criteria listed are specific to TestAmerica. Appendix C also lists the QC requirements for the secondary analyses.



### QAPP Worksheet #13: Secondary Data Criteria and Limitations Table

<b>Secondary Data</b>	<b>Data Source (Originating Organization, Report Title, and Date)</b>	<b>Data Generator(s) (Originating Org., Data Types, Data Generation/Collection Dates)</b>	<b>How Data Will Be Used</b>	<b>Limitations on Data Use</b>
Groundwater Data	GZA, Status Reports, multiple reports: database has been generated through 4 phases of investigation	GZA (TestAmerica), VOC concentration data, multiple events	Historical VOC Concentrations	Generally, analytical methods, sampling SOPs, and the personnel responsible for sample collection remains generally the same as those established for the investigation phases of this project in the original QAPP for continuity and comparability of data sets. Sampling methods changed following the implementation of Revision 3 of the QAPP.  Sampling methods will remain the same after implementation of this Revision 4 of the QAPP.
Biodegradation Parameters Data	GZA, Collected during Sampling Events, Ongoing	GZA (TestAmerica), TOC, dissolved iron, methane, ethane, ethene, pH, DO, ORP, Ongoing	Secondary Lines of Evidence	Sampling and analytical SOPs contained in this QAPP will be used for these parameters. Because these data will be used as secondary lines of evidence, the quality assurance for these parameters will not be as strict as it is for the VOCs.

## QAPP Worksheet #14: Summary of Project Tasks

### **Sampling Tasks:**

1. Wells will continue to be sampled on a semi-annual or biennial frequency depending on the findings at each particular well in accordance with the updated IB Work Plan.
2. Water level measuring will be conducted at the time of the semi-annual sampling. This change from quarterly to semi-annually is in general accordance with the IB Work Plan and Revision 3 of the QAPP.

### **Analysis Tasks:**

1. Monitoring wells will be analyzed for VOCs and biodegradation parameters (dissolved iron, total organic carbon, pH, oxidation-reduction potential (ORP), dissolved oxygen (DO), methane, ethane, and ethane). The parameters pH, ORP, and DO will be measured in the field.

### **Quality Control Tasks:**

1. Implement SOPs for sampling and sample preparation/analysis methods. QC samples are described on Worksheet #26.

### **Secondary Data:**

1. See Worksheet #13.

### **Data Management Tasks:**

1. Analytical data will be input in a database following data validation.

### **Documentation and Records:**

1. See Worksheet #29.
2. Copy of finalized QAPP will be retained in a central file by GZA.

### **Data Packages:**

1. TestAmerica will provide a data package with analytical results for each sampling round. Samples will be run in batches of fewer than 20; however, each round of sampling will be reported as one data package for ease of Puerto Rico data certification and validation.

**Data Review Tasks:**

1. Data review, which includes an in-house examination to check that data have been recorded, transmitted, and processed correctly and data verification, which includes the evaluation of completeness, correctness, and conformance/compliance of a specific data set will be performed by TestAmerica.
2. A Puerto Rico-Certified Chemist will verify that the laboratory data validation report is complete and correct.
3. GZA will perform data validation on the VOC data in accordance with the EPA Region 2 Data Validation SOPs located on the EPA Region 2 webpage (<http://www.epa.gov/region02/qa/documents.htm>) for the VOC data collected for the groundwater monitoring rounds. The criteria for accepting, rejecting, or qualifying data is included in the SOPs. Data validation will not be performed on analytes other than the VOCs because they are being used as secondary evidence only.
4. Validated data and related field logs/notes/records will be reviewed to evaluate overall usability of the data for project purposes. Data limitations will be assessed and data will be compared to Project Quality Objectives (PQOs) and required Action Limits. Corrective action is initiated, as necessary. Final data will be input into database, with the necessary qualifiers, and tables, charts, and graphs are generated. Should reconciliation of the data be needed, it will be accomplished through additional sampling and analysis. The objective of the additional sampling and analysis would be to meet PQOs. Data reconciled would be reported in the appropriate regulatory reporting obligation.

### QAPP Worksheet #15: Reference Limits and Evaluation Table

Matrix: Groundwater  
 Analytical Group: VOCs  
 Concentration Level: Low-High

Analyte	CAS Number	Project Action Limit (ug/L) <sup>1</sup>	Project Quantitation Limit (ug/L) <sup>2</sup>	Analytical Method: 8260B		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs
TCE	79-01-6	5.0	1.0	0.020 ug/L	0.50 ug/L	0.61 ug/L	1.0 ug/L
cis-1,2-DCE	156-59-2	70	1.0	0.060 ug/L	0.50 ug/L	0.65 ug/L	1.0 ug/L
Vinyl Chloride (VC)	75-01-4	0.25	1.0	0.040 ug/L	0.50 ug/L	0.71 ug/L	1.0 ug/L

<sup>1</sup>Project Action Limits are the Puerto Rico Water Quality Standards (PRWQS) or USEPA Maximum Contaminant Level (MCL) if no PRWQS is available.

<sup>2</sup>Project Quantitation Limits are for non-diluted samples and are the Achievable Laboratory Limits QLs.

**QAPP Worksheet #16: Project Schedule/Timeline Table**

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
QAPP Preparation	GZA	8/1/14	4/6/15	QAPP	4/6/15
Intrinsic Biodegradation Study	GZA	8/1/14	4/6/15	Intrinsic Biodegradation Study Work Plan	4/6/15
Monitoring Site Conditions	GZA	Ongoing	Ongoing	Semi-Annual Project Progress Reports	Ongoing

## QAPP Worksheet #17: Sampling Design and Rationale

**Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):**

The sampling approach that will be used at the Site following implementation of this Revision 4 of the QAPP consists of semiannual or biennial sampling of select wells for VOCs and/or biodegradation parameters and semiannual comprehensive groundwater level measurements. The rationale for choosing this approach is based on the Site Conceptual Model and the historical concentrations in the wells.

**Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:**

See Worksheet #18 and attached Site Plan (Figure 1) for details.

**QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements Table**

<b>Sampling Location/ID Number</b>	<b>Matrix</b>	<b>Hydrogeologic Unit</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Number of Samples (identify field duplicates)</b>	<b>Sampling SOP Reference</b>	<b>Rationale for Sampling Location</b>
GZ-519U, OW-101, GZ-503L, GZ-504L, OW-304L, OW-307, WB-3L, WB-4L, GZ-504R, GZ-505R, GZ-506R, IW-1, IW-2, IW-3	Groundwater	See Site Plan	VOCs, dissolved iron, TOC, methane, ethane, DO, ORP, pH	Low-High	18 (14 field samples, 1 duplicate, 1 MS/MSD, 1 equipment blank if non-dedicated pump is used, & 1 trip blank per cooler of VOA vials) sampled semi-annually	3.1.2	Exhibit an apparent increasing temporal trend for [TCE] or are considered source area wells or are being evaluated for the ERD pilot test
GZ-515U, OW-301	Groundwater	See Site Plan	VOCs, dissolved iron, TOC, methane, ethane, DO, ORP, pH	Low-High	2 (2 field samples) sampled biennially	3.1.2	Considered background wells
GZ-504U, GZ-702U, OW-402U, WB-1U, GZ-501L, GZ-701L, OW-101L, WB-1L, WB-2L, GZ-601R, GZ-701R, GZ-702R, GZ-703R, OW-304R, OW-404R, OW-404U, WB-2U	Groundwater	See Site Plan	VOCs, DO, ORP, pH	Low-High	21 (17 field samples, 1 duplicate, 1 MS/MSD, 1 equipment blank if non-dedicated pump is used, & 1 trip blank per cooler of VOA vials) sampled semi-annually	3.1.2	Exhibit an apparent stable or decreasing temporal trend for [TCE]

<b>Sampling Location/ID Number</b>	<b>Matrix</b>	<b>Hydrogeologic Unit</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Number of Samples (identify field duplicates)</b>	<b>Sampling SOP Reference</b>	<b>Rationale for Sampling Location</b>
GZ-501U, GZ-503U, GZ-506U, GZ-511U, OW-105, OW-304U, OW-305U, DEC-204O, GZ-502L, GZ-505L, GZ-601L, OW-1, OW-102, OW-401, OW-402L, OW-403L, OW-404L, OW-405, OW-407, OW-408, BR-308, DEC-203R, OW-402R, OW-305I	Groundwater	See Site Plan	VOCs, DO, ORP, pH		28 (24 field samples, 2 duplicates, 1 MS/MSD, & 1 trip blank per cooler of VOA vials) sampled biennially (every other year)	3.1.2	[TCE] below Puerto Rico Water Quality Standard or exhibit an apparent stable or decreasing temporal trend for [TCE] and have on-Site wells down-gradient of them



**QAPP Worksheet #19: Analytical SOP Requirements Table**

<b>Matrix</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Analytical and Preparation Method/SOP Reference</b>	<b>Sample Volume</b>	<b>Containers (number, size, and type)</b>	<b>Preservation Requirements (chemical, temperature, light protected)</b>	<b>Maximum Holding Time (preparation/analysis)</b>
Groundwater	VOCs	Low/High	8260B+5030/1	120 mL	(3) 40 mL VOA vials	Ice, HCl to pH <2	14 days (7 days if unpreserved)
Groundwater	TOC	Low	SM5310C/2	120 mL	(3) amber 40 mL VOA vials	Ice, H2SO4 to pH <2	28 days
Groundwater	Dissolved Iron	Low	6010B+3005A/5	200 mL	(1) 250 mL plastic	Field filter, then HNO3 to pH <2	6 months
Groundwater	Methane, Ethane, Ethene	Low	RSK-175/6	40 mL	(3) 40 mL VOA vials	Ice, HCl to pH <2	14 days

**QAPP Worksheet #20: Field Quality Control Sample Summary Table**

<b>Matrix</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Analytical and Preparation SOP Reference<sup>4</sup></b>	<b>No. of Sampling Locations</b>	<b>No. of Field Duplicate Pairs</b>	<b>No. of MS/MSD</b>	<b>No. of Field Blanks</b>	<b>No. of Equip. Blanks</b>	<b>No. of Trip Blanks</b>	<b>Total No. of Samples to Lab</b>
Groundwater	VOCs <sup>1</sup>	Low-High	8260B+5030/1	31, 57 <sup>2</sup>	4,6	2,3	1 per day	1 per day <sup>3</sup>	3,5	42,71 <sup>2</sup>
Groundwater	TOC	Low	SM5310C/2	31, 57						31, 54
Groundwater	Dissolved Iron	Low	6010B+3005A/5	14, 16						14, 16
Groundwater	Methane, Ethane, Ethene	Low	RSK-175/6	14, 16						14, 16

<sup>1</sup> One trip blank per cooler of VOA vials will be included for the VOCs.

<sup>2</sup> Semiannual sampling event numbers are shown first. Biennial sampling event numbers are shown second.

<sup>3</sup> One equipment blank per day when non-dedicated equipment is used.

<sup>4</sup> The SOPs referenced above are included in Appendix A of the QAPP.

### QAPP Worksheet #21: Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
1.1.1	Overburden “Machine Operated Hollow-Stem Augering” Boring, 03/08/91, Rev. No. 3	GZA	See SOP in Appendix D	N
1.1.2	Overburden Boring – “Wash and Drive”, 03/08/91, Rev. No. 3	GZA	See SOP in Appendix D	N
1.2.1	Rock Core Drilling, 03/08/91, Rev. No. 3	GZA	See SOP in Appendix D	N
1.2.2	Rotary Drilling, 01/29/86, Rev. No. 2	GZA	See SOP in Appendix D	N
2.1	Well Installations – Overburden Well, 05/15/09, Rev. No. 4	GZA	See SOP in Appendix D	N
2.3	Well Installations – Bedrock Well, 03/08/91, Rev. No. 3	GZA	See SOP in Appendix D	N
2.4	Well Development, 5/29/91	GZA	See SOP in Appendix D	N
3.1.1	Sample Collection – Surface Waters, 03/08/91, Rev. No. 3	GZA	See SOP in Appendix D	N
3.1.2	Sample Collection - Monitoring Wells, 3/2010, Rev. No. 5	GZA	See SOP in Appendix D	N
3.1.7.1	Sample Collection – Surface Soil, 01/29/86, Rev. No. 2	GZA	See SOP in Appendix D	N
3.1.7.2	Sample Collection – Subsurface Soil, 01/29/86, Rev. No. 2	GZA	See SOP in Appendix D	N
3.1.8	Sample Collection – Lake, Pond, and Stream Sediments, 01/29/86, Rev. No. 2	GZA	See SOP in Appendix D	N
3.2.3	Chain-Of-Custody Record Keeping, 03/23/87, Rev. No. 1	GZA	See SOP in Appendix D	N

**QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table**

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference <sup>1</sup>
Photo-Ionization Detector: MiniRAE 2000	Calibrate with Manufacturer-Supplied Standard	Battery Check Inspect Vapor Element			Daily	+/- 10%	Re-analyze standard. If it is still outside the acceptance criteria then replace with a different meter.	GZA	MiniRAE 2000 Operation and Maintenance Manual
Electronic Water Level Indicator: Slope Indicator		Battery Check			Daily			GZA	Water Level Indicator Instruction Sheet
Water Quality Meter: In-Situ SmarTROLL	Calibrate with Manufacturer-Supplied Standards				Weekly	+/- 10%	Re-analyze standard. If it is still outside the acceptance criteria then replace with a different meter.	GZA	In-Situ SmarTROLL Operator 's Manual
		Battery Check Inspect Temperature, Conductivity, and pH Elements			Daily				
Vibrating Wire Readout Box: Geokon GK-403	Send to Manufacturer for Calibration				Yearly			GZA	GK-403 Vibrating Wire Readout Box Instruction Manual
Single Channel Datalogger: Geokon LC-2		Battery Check			Semi-Annually			GZA	Model LC-2 Single Channel Datalogger Instruction Manual

<b>Field Equipment</b>	<b>Calibration Activity</b>	<b>Maintenance Activity</b>	<b>Testing Activity</b>	<b>Inspection Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Responsible Person</b>	<b>SOP Reference<sup>1</sup></b>
Proactive Tempest/Twister Pump		Inspect Connections			Before Use				Proactive Pump & Controller Brochure

<sup>1</sup> Equipment User Manuals can be found in Appendix E.

**QAPP Worksheet #23: Analytical SOP References Table**

<b>Reference Number</b>	<b>Title, Revision Date, and/or Number</b>	<b>Definitive or Screening Data</b>	<b>Analytical Group</b>	<b>Instrument</b>	<b>Organization Performing Analysis</b>	<b>Modified for Project Work? (Y/N)</b>
1	SA-VO-004, Rev. 1: Volatile Compounds by GC/MS (8260B)	Definitive	VOCs	Agilent GC/MS	TestAmerica Savannah	N
2	SA-GE-204, Rev. 4: Carbon Content in Water: Total Organic Carbon (TOC, SM 5310B)	Definitive	TOC	Tekmar Phoenix 8000	TestAmerica Savannah	N
3	SA-ME-050, Rev. 15: Liquid Preparation Procedures for ICP and ICP/MS AND SA-ME-070, Rev. 16: ICP Analysis (Methods 200.7 and 6010B)	Preparation/ Definitive	Dissolved Iron	TJA 61E OR Varian ICP	TestAmerica Savannah	N
4	SA-VO-007, Rev. 2: Dissolved Gases in Water (RSK-175)	Definitive	Methane, Ethane, Ethene	Agilent GC w. FID/TCD	TestAmerica Savannah	N
3.1.2	Sample Collection - Monitoring Wells, 3/2010, Rev. No. 5	Definitive	DO, ORP, pH	In-Situ SmartTroll	GZA	N

<sup>1</sup>Laboratory SOPs are located in Appendix A of the QAPP.

**QAPP Worksheet #24: Analytical Instrument Calibration Table**

<b>Instrument</b>	<b>Calibration Procedure</b>	<b>Frequency of Calibration</b>	<b>Acceptance Criteria</b>	<b>Corrective Action (CA)</b>	<b>Person Responsible for CA</b>	<b>SOP Reference<sup>1</sup></b>
Agilent GC/MS	6 – 9 point cal curve	As needed/6 months	RSD <20% or cc>0.990 + ICV 70-130%; CCV 80-120%	Reanalyze curve; reanalyze ICV	Analyst	1
Tekmar Phoenix 8000	6 point cal curve	As needed/ monthly	Linear, cc >0.990 + ICV 70-130%; CCV 90-110%	Reanalyze curve; reanalyze ICV	Analyst	2
TJA 61E OR Varian ICP	2 point cal (zero and high)	Each day/each run	Readback of high std. 95-105%+ICV 90-110%; CCV90-110%	Reanalyze curve; reanalyze high std./ICV	Analyst	3
Agilent GC w. FID/TCD	7-10 point cal curve	As needed	RSD <25% or cc>0.990+ICV 75-125%; CCV 75-125%	Reanalyze curve; reanalyze ICV	Analyst	4

## QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference <sup>1</sup>
Agilent GC/MS	1. replace column 2. add/change pump oil 3. clean source 4. replace trap 5. replace gas cylinder	N/A	6. inspect injection port septa, sleeve, and liner, change as needed 7. inspect autosample tubing and syringe, replace as needed	1. as needed 2. as needed 3. as needed 4. as needed 5. at 500 psi 6. weekly 7. weekly	N/A	N/A	TestAmerica Analyst	1
Tekmar Phoenix 8000	1. replace catalyst 2. fill humidifier	N/A	3. inspect detector windows, clean as needed 4. inspect syringe, clean or replace as needed	1. as needed 2. as needed 3. weekly 4. monthly or as needed	N/A	N/A	TestAmerica Analyst	2
TJA 61E OR Varian ICP	1. rotate pump tubing, replace as needed 2. clean nebulizer	N/A	3. inspect filters, clean or replace as needed 4. inspect torch tip, clean or replace as needed	1. every other day 2. as needed 3. monthly 4. weekly	N/A	N/A	TestAmerica Analyst	3
Agilent GC w. FID/TCD	1. replace column 2. clean detectors	N/A	3. inspect injection port, change septa, sleeve, liner as needed 4. inspect autosampler lines and syringe, clean or replace as needed	1. as needed 2. as needed 3. daily 4. weekly	N/A	N/A	TestAmerica Analyst	4

<sup>1</sup>See Appendix A for SOPs.



## QAPP Worksheet #26: Sample Handling System

<b>SAMPLE COLLECTION, PACKAGING, AND SHIPMENT</b>
Sample Collection (Personnel/Organization): James Roehrig/GZA
Sample Packaging (Personnel/Organization): James Roehrig/GZA
Coordination of Shipment (Personnel/Organization): James Roehrig/GZA & Jess Hornsby/TestAmerica
Type of Shipment/Carrier: Air/FedEx
<b>SAMPLE RECEIPT AND ANALYSIS</b>
Sample Receipt (Personnel/Organization): Jess Hornsby /TestAmerica
Sample Custody and Storage (Personnel/Organization): Jess Hornsby /TestAmerica
Sample Preparation (Personnel/Organization): Jess Hornsby /TestAmerica
Sample Determinative Analysis (Personnel/Organization): Jess Hornsby /TestAmerica
<b>SAMPLE ARCHIVING</b>
Field Sample Storage (No. of days from sample collection): 30 days after the final report is issued
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 30 days after the final report is issued
Biological Sample Storage (No. of days from sample collection): N/A
<b>SAMPLE DISPOSAL</b>
Personnel/Organization: Jess Hornsby /TestAmerica
Number of Days from Analysis: Unconsumed sample material will be discarded in an environmentally sound manner by the laboratory two weeks after holding times for the assigned analyses have expired. TestAmerica will dispose of samples, sample extracts and digestates 30 days after the final report is issued.

## QAPP Worksheet #27: Sample Custody Requirements

### **Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):**

Sample collection will be conducted in accordance with the appropriate SOP. Sample custody will be documented to support the data quality objectives. Samples will be packed into a cooler with frozen "blue ice" or ice cubes for delivery to the laboratory. The cooler will be transported to the laboratory by a courier, using custody seals.

### **Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):**

The samples will be inspected by the Laboratory Sample Controller, or other qualified laboratory personnel. The Sample Receipt Checklist is used to document the receipt of the samples and includes a check for breakage, correct container and preservative, temperature of the cooler, holding times, and for other factors that may affect quality. The samples will be compared to their description on the COC form. Discrepancies in the number or the designations of the samples will be noted on the form, brought to the attention of the GZA Project Manager and resolved at the site team's instruction. The COC form will be signed and the date and time recorded to formally accept the samples into laboratory custody.

The sampling date of each sample will be recorded on the COC by the sampler. The sample holding times (see Worksheet #19) will be checked to demonstrate that the samples can be extracted and/or analyzed within the appropriate holding times.

A copy of the COC form will be sent to the GZA Project Manager by the laboratory. The original COC form will be retained in laboratory files. Additional COC copies will be retained by the laboratory.

Once samples have been labeled with unique laboratory identification numbers, they will be placed in the refrigerator. Refrigerator temperatures will be maintained at 1.5 to 6° Celsius and will be monitored twice each business day.

The laboratory will file the original COC form, the raw data, and a copy of the analytical report.

Unconsumed sample material will be discarded in an environmentally sound manner by the laboratory two weeks after holding times for the assigned analyses have expired. TestAmerica will dispose of samples, sample extracts and digestates 30 days after the final report is issued.

### **Sample Identification Procedures:**

Labels will be affixed to the sample containers with the following information: sample number, sample location, date/time, and name of sampler.

### **Chain-of-custody Procedures:**

Samples will be tracked through collection, shipment, and laboratory receipt by a COC form. The COC form will be signed by the individuals who handle the samples. The original COC accompanies the samples until the project is complete and is kept in a permanent project file. A copy of the COC is also kept with the GZA Project Manager, the laboratory manager, and attached to the data package. An example COC, shipping order, and custody seal are included in Appendix B.

**QAPP Worksheet #28: QC Samples Table**

Matrix	Groundwater				
Analytical Group	VOCs				
Concentration Level	Low/High				
Sampling SOP	3.2.1				
Analytical Method/ SOP Reference	8260B/1				
Field Sampling Organization	GZA				
Analytical Organization	TestAmerica Savannah				
No. of Sample Locations	57				
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits (Measurement Performance Criteria)	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)
Method Blank	1/20 field samples	<MDL	Reanalyze associated samples	TestAmerica Analyst	Accuracy
LCS/LCSD	1 set/20 field samples	70-130% RPD < 20%	Reanalyze associated samples	TestAmerica Analyst	Accuracy Precision
MS/MSD	1 set/20 field samples	70-130% RPD < 20%	Flag parent sample and narrate	TestAmerica Analyst/Project Manager	Accuracy Precision
Field Duplicate	1 set/10 field samples	RPD < 20%	Flag parent sample and narrate	GZA GeoEnvironmental, Inc. Project QA Officer	Precision
Initial Calibration	When %D of CCVS has gross exceedance	$\leq 20\%RSD$ ; $RRF > 0.050$	Flag parent sample and narrate	TestAmerica Analyst	Accuracy
Continuing Calibration Verification Standard	Every 12 hours before analysis of method blanks and samples	$\%D \leq 20\%$	Flag parent sample and narrate	TestAmerica Analyst	Accuracy
Surrogates	All samples, blanks, LCS, MS	80-120%	Flag parent sample and narrate	TestAmerica Analyst	Accuracy

<b>QC Sample</b>	<b>Frequency/ Number</b>	<b>Method/SOP QC Acceptance Limits (Measurement Performance Criteria)</b>	<b>Corrective Action</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>
Internal Standard	All samples and standards	<30 second change in retention time compared with last initial calibration; <factor of 2 (-50% to +100%) change in internal standard area compared with last initial calibration	Reanalyze associated samples	TestAmerica Analyst	Accuracy/Bias

### QAPP Worksheet #29: Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes	Sample Receipt, Custody, and Tracking Records	Sample Receipt, Custody, and Tracking Records	Data Validation Reports (including data package completeness check)	Status Report
Chain-of-Custody Records	Equipment Calibration Logs	Equipment Calibration Logs	Corrective Action Forms	
Air Bills	Equipment Maintenance, Testing, and Inspection Logs	Equipment Maintenance, Testing, and Inspection Logs		
Custody Seals	Sampling & Field Parameters Logs	Sample Prep Logs		
Field Audit Checklist <sup>1</sup> (including field sampling)	Field Audit Checklist <sup>1</sup> (including field analysis)	Reported Field Sample Results		
	Corrective Action Forms	Lab Reports (Reported Results for Standards, QC Checks, and QC Samples, sample disposal records and raw data)		
		Corrective Action Forms		

<sup>1</sup> See Appendix B for example field audit checklist.

**QAPP Worksheet #30: Analytical Services Table**

<b>Matrix</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Sample Locations/ID Numbers</b>	<b>Analytical SOP</b>	<b>Data Package Turnaround Time</b>	<b>Laboratory/Organization (Name and Address, Contact Person and Telephone Number)</b>	<b>Backup Laboratory/ Organization (Name and Address, Contact Person and Telephone Number)</b>
Groundwater	VOCs	Low/High	See QAPP Worksheet #18	SA-VO-004, Rev. 1: Volatile Compounds by GC/MS (8260B)	28 Days	TestAmerica Laboratories, Inc.; Jess Hornsby 813-885-7427	N/A
Groundwater	TOC	Low	See QAPP Worksheet #18	SA-GE-204, Rev. 4: Carbon Content in Water: Total Organic Carbon (TOC)	28 Days	TestAmerica Laboratories, Inc.; Jess Hornsby 813-885-7427	N/A
Groundwater	Dissolved Iron	Low	See QAPP Worksheet #18	SA-ME-050, Rev. 15: Liquid Preparation Procedures for ICP and ICP/MS AND SA-ME-070, Rev. 16: ICP Analysis (Methods 200.7 and 6010B)	28 Days	TestAmerica Laboratories, Inc.; Jess Hornsby 813-885-7427	N/A
Groundwater	Methane, Ethane, Ethene	Low	See QAPP Worksheet #18	SA-VO-007, Rev. 2: Dissolved Gases in Water (RSK-175)	28 Days	TestAmerica Laboratories, Inc.; Jess Hornsby 813-885-7427	N/A



**QAPP Worksheet #31: Planned Project Assessments Table**

<b>Assessment Type</b>	<b>Frequency</b>	<b>Internal or External</b>	<b>Organization Performing Assessment</b>	<b>Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)</b>	<b>Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)</b>
Data review	Each test	Internal	TestAmerica Savannah	Analyst, department manager; TA Savannah	Analyst, department manager; TA Savannah	Analyst, department manager; TA Savannah	QA manager; TA Savannah
Report review	Each report	Internal	TestAmerica Savannah	Project manager; TA Savannah	Department manager; TA Savannah	Project manager, department manager; TA Savannah	QA manager, project manager; TA Savannah
Department audits	Annually	Internal	TestAmerica Savannah	QA manager; TA Savannah	Department manager; TA Savannah	Department manager; TA Savannah	QA manager; TA Savannah
Data Usability	Each sampling event	External	GZA	Chunhua Liu, Risk Assessor; GZA	John Colbert, Project Manager, GZA	John Colbert, Project Manager, GZA	John Colbert, Project Manager, GZA
Field Audit	Annually	Internal	GZA	James Roehrig, Project Engineer	John Colbert, Project Manager, GZA	John Colbert, Project Manager, GZA	John Colbert, Project Manager, GZA

### QAPP Worksheet #32: Assessment Findings and Corrective Action Responses

<b>Assessment Type</b>	<b>Nature of Deficiencies Documentation</b>	<b>Individual(s) Notified of Findings (Name, Title, Organization)</b>	<b>Timeframe of Notification</b>	<b>Nature of Corrective Action Response Documentation</b>	<b>Individual(s) Receiving Corrective Action Response (Name, Title, Org.)</b>	<b>Timeframe for Response</b>
Data Usability	Written Audit Report	John Colbert, Project Manager, GZA	48 hours after audit	Letter	Jess Hornsby Project Manager, TestAmerica & James Roehrig, Sampling Team Leader, GZA	24 hours after notification
Field Audit	Written Audit Report	John Colbert, Project Manager, GZA	48 hours after audit	Letter	James Roehrig, Sampling Team Leader, GZA	24 hours after notification

### QAPP Worksheet #33: QA Management Reports Table

<b>Type of Report</b>	<b>Frequency (daily, weekly monthly, quarterly, annually, etc.)</b>	<b>Projected Delivery Date(s)</b>	<b>Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)</b>	<b>Report Recipient(s) (Title and Organizational Affiliation)</b>
Data Usability Report	Each Sampling Event	Within 4 weeks of receipt of data from field and laboratory.	Chunhua Liu, Risk Assessor; GZA	John Colbert, Project Manager, GZA
Field Audit	Annually	Within 4 weeks of completion of field work	James Roehrig, Project Engineer; GZA	John Colbert, Project Manager, GZA

**QAPP Worksheet #34: Verification (Step I) Process Table**

<b>Verification Input</b>	<b>Description</b>	<b>Internal/ External</b>	<b>Responsible for Verification (Name, Organization)</b>
Chain-of-custody and shipping forms	The samples will be inspected by the Laboratory Sample Controller, or other qualified laboratory personnel. The Sample Receipt Checklist is used to document the receipt of the samples and includes a check for breakage, correct container and preservative, temperature of the cooler, holding times, and for other factors that may affect quality.	Internal	Laboratory Sample Controller TestAmerica
Audit Reports	<p>Performance audits may consist of blind samples, or split samples with another laboratory. Internal performance audits consist of a Laboratory QA Officer ordering blind samples and double blind samples for the laboratory as the need arises. These samples are used for internal purposes only. The scores are discussed with the appropriate Laboratory Supervisor. The Laboratory's Corporate QA Manager performs an annual audit each year to determine if the procedures implemented by the TestAmerica divisions are in compliance with the QA plan and the SOPs.</p> <p>Upon report completion, a copy of the audit reports will be placed in the site file. If corrective actions are required, a copy of the documented corrective action taken will be attached to the appropriate audit report in the site file. Site file audit reports will be reviewed periodically internally to check that that all appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, the site manager will be notified so that action is taken.</p>	<p>Internal</p> <p>External</p>	<p>Jess Hornsby TestAmerica</p> <p>John Colbert GZA</p>
Field Notes	Field notes will be reviewed by GZA and kept in the site file.	Internal	John Colbert GZA

<b>Verification Input</b>	<b>Description</b>	<b>Internal/ External</b>	<b>Responsible for Verification (Name, Organization)</b>
Laboratory Data	<p>The laboratory will prepare a data validation report. A Puerto Rico-Certified Chemist will verify that the laboratory data validation report is complete and correct.</p> <p>The laboratory's data validation information will be reviewed and GZA will perform an independent data validation on the VOC data in accordance with the EPA Region 2 Data Validation SOPs located on the EPA Region 2 webpage (<a href="http://www.epa.gov/region02/qa/documents.htm">http://www.epa.gov/region02/qa/documents.htm</a>) for the VOC data collected for the groundwater monitoring rounds. The criteria for accepting, rejecting, or qualifying data is included in the SOPs. Data validation will not be performed on analytes other than the VOCs because they are being used as secondary evidence only</p>	<p>Internal External</p> <p>External</p>	<p>Jess Hornsby TestAmerica Daliz M. Estades Santaliz</p> <p>Chunhua Liu, GZA</p>

**QAPP Worksheet #35: Validation (Steps IIa and IIb) Process Table**

<b>Step IIa/IIb</b>	<b>Validation Input</b>	<b>Description</b>	<b>Responsible for Validation (Name, Organization)</b>
IIb	Onsite analytical work	Onsite analytical data will be reviewed against QAPP requirements for completeness and accuracy based on the field calibration records and field notes.	John Colbert, GZA
IIa	SOPs	Check that sampling and analytical SOPs were followed.	John Colbert, GZA
IIa/IIb	Documentation of Method QC Results	Establish that the method required QC samples were run and met required limits.	Jess Hornsby, TestAmerica
IIb	Project Quantitation Limits	Check that sample results met the project quantitation limits	Chunhua Liu, GZA



**QAPP Worksheet #36: Validation (Steps IIa and IIb) Summary Table**

<b>Step IIa/IIb</b>	<b>Matrix</b>	<b>Analytical Group</b>	<b>Concentration Level</b>	<b>Validation Criteria</b>	<b>Data Validator (title and organizational affiliation)</b>
IIa	Groundwater	VOCs	Low/Medium	Puerto Rico Certified Chemist	Daliz M. Estades Santaliz
IIb	Groundwater	VOCs	Low/Medium	Region 2 Data Validation Guidance	Chunhua Liu, GZA

### QAPP Worksheet #37: Usability Assessment

Data validation will be performed for VOC data generated for the project. Data generated for biodegradation parameters, from either the field or from the laboratory, will not undergo data validation because they will be used as secondary evidence only, and do not require a formal data validation.

**Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:**

The data usability assessment is an evaluation based on the results of data quality in the context of the overall project decisions or objectives. A usability assessment evaluates whether data meet project quality objectives as they relate to the decision to be made, and evaluates whether data are suitable for making that decision. All types of definitive data (e.g., sampling, on-site analytical, off-site laboratory) are relevant to the usability assessment. The usability assessment is the final step of data review and can be performed only on data of known and documented quality.

The following items will be assessed and conclusions drawn based on their results:

**Precision** – Results of laboratory and laboratory duplicates, sample and field duplicates, laboratory control sample (LCS) and laboratory control sample duplicate (LCSD), and matrix spike (MS) and matrix spike duplicate (MSD) will be evaluated. For each duplicate pair, the relative percent difference (RPD) will be calculated for each analyte when the original and duplicate values are greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented in this QAPP (Worksheet #12). A discussion will follow summarizing the results of the laboratory and field precision. Conclusions about the precision of the analyses will be drawn and limitations on the use of the data will be described. Calculated RPDs (in percentage) are only applicable when the sample values are greater than or equal to two times the respective analytical reporting limits (RLs). For the primary and duplicate sample results that are less than two times the respective analytical RL, the precision goal is met when the absolute difference (AD) between the results is less than two times the RL.

**Accuracy** – Results for laboratory method blanks, instrument blanks, trip blanks, equipment blanks, and other appropriate QA/QC data (e.g., surrogate recoveries and internal standard results) will be evaluated and the results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. A discussion will follow summarizing the results of the laboratory accuracy. Conclusions about the accuracy of the analyses will be drawn and limitations on the use of the data will be described.

**Sensitivity** – Results for laboratory fortified blanks will be evaluated and the results for each analyte will be checked against the measurement performance criteria presented on Worksheet #12. Results for analytes that exceed criteria will be identified. A discussion will follow summarizing the results of the laboratory sensitivity. Conclusions about the sensitivity of the analyses will be drawn and limitations on the use of the data will be described.

**Representativeness** – Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is achieved through proper development of the field sampling program. The sampling program must be designed so that the samples collected are as representative as possible of the medium being sampled and that a sufficient number of samples will be collected.

**Comparability** – The results of this study will be used as a benchmark for assessing comparability for data collected during potential future

sampling events using the same or similar sampling and analytical SOPs.

**Completeness** – Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data are complete and valid if they meet all acceptance criteria including accuracy, precision, and other criteria specified by the particular analytical method being used. Data with minor exceedances in accuracy and precision may be considered usable based on a data usability assessment. Field completeness will be determined by the GZA Project Manager and will be based on Site conditions and conditions of the monitoring wells at the time of sampling. The goal is greater than 90 percent and is calculated as follows:

$$\% FC = (A/P) \times 100$$

where,

%FC = Field Percent Completeness;

A = Actual number of samples collected; and

P = Number of planned samples to collect.

Laboratory completeness will be estimated as the percentage of usable measurements and calculated as follows:

$$\% C = (U/T) \times 100$$

where:

%C = Percent completeness;

U = Number of measurements judged usable or not able to analyze; and

T = Total number of measurements.

Unless otherwise specified by the QAPP, the goal for laboratory completeness is 90 percent.

**Reconciliation** – As part of the Data Validation, each of the PQOs presented on Worksheet #12 will be examined to evaluate if the objective was met. This exam will include a combined overall assessment of the results of each analysis pertinent to an objective. The final report will include a summary of the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and limitations on the usability of the data will be described.

**Describe the evaluative procedures used to assess overall measurement error associated with the project:**

The results for the LCS/LCSD and MS/MSD will be compared to the requirements listed on Worksheet #12. A discussion will follow summarizing overall accuracy/bias. Conclusions about the overall accuracy/bias of the analyses will be drawn and limitations on the use of the data will be described.

**Identify the personnel responsible for performing the usability assessment:**

The Data Usability Assessment will be performed by a team of personnel at GZA. The QA Officer will be responsible for the Usability Assessment. She will also be responsible for assigning task work to the individual task members who will be supporting the Data Usability Assessment. The QA Officer will review at least 20% of the data validated by the trained personnel and is responsible to oversee the whole data validation process.

**Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:**

A data usability assessment report will be submitted to summarize the usability of the validated data. The report will include:

- A summary of data validation results in text and table formats,
- Overall data usability and completeness,
- Evaluation of each data quality indicator (whether meet the criteria, what potential impacts on data usability),
- Deviations (e.g., holding time, QC performance criteria, sample location, sample collection SOPs) from the SAP and/or the QAPP and the impact of deviations on the usability of data,
- Problems with documentation or custody procedures and the impact on the usability of data,
- Damaged samples and the usability of the associated data, and
- Other relevant issues.

## **FIGURES**







**APPENDIX A**  
**LABORATORY STANDARD OPERATING PROCEDURES (TESTAMERICA)**

# Quality Assurance Manual

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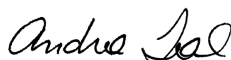
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Laboratory Director  
Todd Baumgartner

July 3, 2014

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Date



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EH&S Coordinator  
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**SECTION 2. TABLE OF CONTENTS**

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
-	COVER PAGE	V1M2 Sec. 4.2.8.3		1
1.0	TITLE PAGE			2
2.0	TABLE OF CONTENTS	V1M2 Secs. 4.2.8.3-4.2.8.4		3
3.0	INTRODUCTION, SCOPE, AND APPLICABILITY	V1M2 Sec. 4.2.8.4		14
3.1	Introduction And Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	14
3.2	Terms And Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	14
3.3	Scope / Fields Of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	15
3.4	Management Of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	15
4.0	MANAGEMENT REQUIREMENTS	V1M2 Sec. 4		17
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	17
4.2	Roles And Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	17
4.3	Deputies	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	21
5.0	QUALITY SYSTEM			24
5.1	Quality Policy Statement	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	24
5.2	Ethics And Data Integrity	V1M2 Secs. 4.1.5; 4.1.6; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	24
5.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	25
5.4	QA/QC Objectives For The Measurement Of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	25
5.5	Criteria For Quality Indicators			27
5.6	Statistical Quality Control			28
5.7	Quality System Metrics			28
6.0	DOCUMENT CONTROL	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	29
6.1	Overview			29
6.2	Document Approval And Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	29

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
6.3	Procedures For Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	30
6.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	30
7.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	31
7.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	31
7.2	Review Sequence And Key Personnel	V1M2 Sec. 4.4.5	4.4.5	32
7.3	Documentation	V1M2 Sec. 5.7.1	5.7.1	32
7.4	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	33
7.5	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	34
7.6	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	34
7.7	Client Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	34
8.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	35
8.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	35
8.2	Qualifying And Monitoring	V1M2 Secs. 4.5.1; 4.5.2; 4.5.3; 4.5.5	4.5.1; 4.5.2; 4.5.3	35
8.3	Oversight And Reporting	V1M2 Sec. 4.5.5		37
8.4	Contingency Planning			38
9.0	PURCHASING SERVICES AND SUPPLIES	V1M2 Sec. 4.6.1	4.6.1	39
9.1	Overview	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	39
9.2	Glassware	V1M2 Sec. 5.5.13.1		39
9.3	Reagents, Standards & Supplies	V1M2 Secs. 4.6.2; 4.6.3; 4.6.4	4.6.2; 4.6.3; 4.6.4	39
9.4	Purchase Of Equipment / Instruments / Software			41
9.5	Services			41
9.6	Suppliers			41
10.0	COMPLAINTS	V1M2 Sec. 4.8	4.8	43
10.1	Overview			43
10.2	External Complaints			43
10.3	Internal Complaints			44
10.4	Management Review			44
11.0	CONTROL OF NON-CONFORMING WORK	V1M2 Secs. 4.9.1; 5.10.5	4.9.1; 5.10.5	45
11.1	Overview	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	45

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
11.2	Responsibilities And Authorities	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5; 5.2.7	4.9.1; 4.11.3; 4.11.5	45
11.3	Evaluation Of Significance And Actions Taken	V1M2 Secs. 4.9.1; 4.11.3; 4.11.5	4.9.1; 4.11.3; 4.11.5	46
11.4	Prevention Of Nonconforming Work	V1M2 Secs. 4.9.4; 4.11.2	4.9.2; 4.11.2	46
11.5	Method Suspension / Restriction (Stop Work Procedures)	V1M2 Secs. 4.9.1; 4.9.2; 4.11.5	4.9.1; 4.9.2; 4.11.5	46
12.0	CORRECTIVE ACTION	V1M2 Sec. 4.11		48
12.1	Overview	V1M2 Secs. 4.9.2; 4.11.1; 4.11.2	4.9.2; 4.11.1; 4.11.2	48
12.2	General	V1M2 Sec. 4.11.2; 4.11.3	4.11.2; 4.11.3	48
12.3	Closed Loop Corrective Action Process	V1M2 Sec. 4.11.2; 4.11.3; 4.11.4; 4.11.6; 4.11.7; 4.12.2	4.11.2; 4.11.3; 4.11.4; 4.12.2	49
12.4	Technical Corrective Actions	V1M2 Sec. 4.11.6		50
12.5	Basic Corrections	V1M2 Secs. 4.11.1; 4.13.2.3	4.11.1; 4.13.2.3	51
13.0	PREVENTIVE ACTION / IMPROVEMENT	V1M2 Secs. 4.10; 4.12.1; 4.12.2	4.10; 4.12.1; 4.12.2	56
13.1	Overview	V1M2 Secs. 4.15.1; 4.15.2	4.15.1; 4.15.2	56
14.0	CONTROL OF RECORDS	V1M2 Secs. 4.2.7; 4.13.1.1; 4.13.3	4.2.7; 4.13.1.1	58
14.1	Overview	V1M2 Secs. 4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3; 4.13.3	4.13.1.1; 4.13.1.2; 4.13.1.3; 4.13.1.4; 4.13.2.1; 4.13.2.2; 4.13.2.3	58
14.2	Technical And Analytical Records	V1M2 Sec. 4.13.2.2 - 4.13.2.3	4.13.2.2; 4.13.2.3	62
14.3	Laboratory Support Activities			63
14.4	Administrative Records			63
14.5	Records Management, Storage And Disposal	V1M2 Sec. 4.13.3		64
15.0	AUDITS			65
15.1	Internal Audits	V1M2 Sec. 4.2.8.1; 4.14; 4.14.1; 4.14.2; 4.14.3; 4.14.5; 5.9.1; 5.9.2	4.14.1; 4.14.2; 4.14.3; 5.9.1; 5.9.2	65
15.2	External Audits	V1M2 Secs. 4.14.2; 4.14.3	4.14.2; 4.14.3; 4.14.4	67



Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
15.3	Audit Findings	V1M2 Secs. 4.14.2; 4.14.3; 4.14.5		67
16.0	MANAGEMENT REVIEWS	V1M2 Sec. 4.1.6; 4.15; 4.15.1; 4.15.2	4.1.6; 4.15.1; 4.15.2	69
16.1	Quality Assurance Report			69
16.2	Annual Management Review	V1M2 Sec. 4.2.2; 4.15.3	4.2.2	69
16.3	Potential Integrity Related Managerial Reviews			70
17.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	71
17.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	71
17.2	Education And Experience Requirements For Technical Personnel	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	71
17.3	Training	V1M2 Sec. 5.2.5	5.2.5	73
17.4	Data Integrity And Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		74
18.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		76
18.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	76
18.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	76
18.3	Work Areas	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	77
18.4	Floor Plan			76
18.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	77
19.0	TEST METHODS AND METHOD VALIDATION	V1M2 Sec. 5.4.1	5.4.1	78
19.1	Overview	V1M2 Sec. 5.4.1	5.4.1; 5.4.5.1	78
19.2	Standard Operating Procedures (Sop)	V1M2 Secs. 4.2.8.5; 4.3.3.1; 5.4.2	4.3.3.1; 5.4.2	78
19.3	Laboratory Methods Manual	V1M2 Sec. 4.2.8.5		78

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
19.4	Selection Of Methods	V1M2 Secs. 4.13.3; 5.4.1; 5.4.2; 5.4.3. V1M4 Secs. 1.4; 1.5.1; 1.6.1; 1.6.2; 1.6.2.1; 1.6.2.2	5.4.1; 5.4.2; 5.4.3; 5.4.4; 5.4.5.1; 5.4.5.2; 5.4.5.3	79
19.5	Laboratory Developed Methods And Non-Standard Methods	V1M2 Sec. 5.4.2. V1M4 Sec. 1.5.1	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.6	Validation Of Methods	V1M2 Sec. 5.4.2. V1M4 Secs. 1.5.1; 1.5.2; 1.5.2.1; 1.5.2.2; 1.5.3	5.4.2; 5.4.4; 5.4.5.2; 5.4.5.3	81
19.7	Method Detection Limits (mdl) / Limits Of Detection (LOD)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.5.2; 1.5.2.1; 1.5.2.2	5.4.5.3	83
19.8	Instrument Detection Limits (Idl)	V1M2 Sec. 5.9.3		83
19.9	Verification Of Detection And Reporting Limits	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.2.1		84
19.10	Retention Time Windows	V1M2 Sec. 5.9.3		84
19.11	Evaluation Of Selectivity	V1M2 Sec. 5.9.3. V1M4 Sec. 1.5.4; 1.7.3.6		84
19.12	Estimation Of Uncertainty Of Measurement	V1M2 Sec. 5.1.1; 5.1.2; 5.4.6	5.1.1; 5.1.2; 5.4.6.1; 5.4.6.2; 5.4.6.3	84
19.13	Sample Reanalysis Guidelines	V1M2 Sec 5.9.1	5.9.1	85
19.14	Control Of Data	V1M2 Secs. 5.4.7.1; 5.4.7.2; 5.9.1	5.4.7.1; 5.4.7.2; 5.9.1	86
20.0	EQUIPMENT AND CALIBRATIONS	V1M2 Secs. 5.5.4; 5.5.5; 5.5.6	5.5.4; 5.5.5; 5.5.6; 5.6.1	93
20.1	Overview	V1M2 Secs. 5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10	5.5.1; 5.5.2; 5.5.3; 5.5.5; 5.5.10; 5.6.1	93
20.2	Preventive Maintenance	V1M2 Secs. 5.5.1; 5.5.3; 5.5.7; 5.5.9	5.5.1; 5.5.3; 5.5.7; 5.5.9; 5.6.1	93
20.3	Support Equipment	V1M2 Secs. 5.5.10; 5.5.11; 5.5.13.1	5.5.10; 5.5.11; 5.6.2.1.2; 5.6.2.2.1; 5.6.2.2.2	94
20.4	Instrument Calibrations	V1M2 Secs. 5.5.8; 5.5.10; 5.6.3.1. V1M4 Sec. 1.7.1.1; 1.7.2	5.5.8; 5.5.9; 5.5.10; 5.6.1; 5.6.2; 5.6.3.1	96
20.5	Tentatively Identified Compounds (TICS) – GC/MS Analysis			99

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
20.6	Gc/Ms Tuning			99
21.0	MEASUREMENT TRACEABILITY			104
21.1	Overview	V1M2 Sec. 5.6.3.1	5.6.2.1.2; 5.6.2.2.2; 5.6.3.1	104
21.2	NIST-Traceable Weights And Thermometers	V1M2 Secs. 5.5.13.1; 5.6.3.1; 5.6.3.2	5.6.3.1; 5.6.3.2	104
21.3	Reference Standards / Materials	V1M2 Secs. 5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.6.4.1; 5.6.4.2; 5.9.1; 5.9.3	5.6.3.1; 5.6.3.2; 5.6.3.3; 5.6.3.4; 5.9.1	104
21.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	V1M2 Secs. 5.6.4.2; 5.9.3		105
22.0	SAMPLING			108
22.1	Overview	V1M2 Secs. 5.7.1; 5.7.3	5.7.1; 5.7.3	108
22.2	Sampling Containers			108
22.3	Definition Of Holding Time			108
22.4	Sampling Containers, Preservation Requirements, Holding Times			108
22.5	Sample Aliquots / Subsampling	V1M2 Sec. 5.7.1	5.7.1	108
23.0	HANDLING OF SAMPLES	V1M2 Sec. 5.8.1	5.8.1	110
23.1	Chain Of Custody (COC)	V1M2 Secs. 5.7.2; 5.7.4; 5.8.4; 5.8.7.5; 5.8.8; 5.9.1	5.7.2; 5.8.4; 5.9.1	110
23.2	Sample Receipt	V1M2 Secs. 5.8.1; 5.8.2; 5.8.3; 5.8.5; 5.8.7.3; 5.8.7.4; 5.8.7.5	5.8.2; 5.8.3	111
23.3	Sample Acceptance Policy	V1M2 Secs. 5.8.6; 5.8.7.2		112
23.4	Sample Storage	V1M2 Secs. 5.7.4; 5.8.4	5.8.4	113
23.5	Hazardous Samples And Foreign Soils			113
23.6	Sample Shipping	V1M2 Sec. 5.8.2	5.8.2	114
23.7	Sample Disposal			114
24.0	ASSURING THE QUALITY OF TEST RESULTS			119
24.1	Overview	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119
24.2	Controls	V1M2 Secs. 5.9.2; 5.9.3	5.9.2	119

Sec. No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
24.3	Negative Controls	V1M2 Secs. 5.9.2; 5.9.3 V1M4 Secs. 1.7.3; 1.7.3.1; 1.7.4.1	5.9.2	119
24.4	Positive Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. 1.7.3; 1.7.3.2; 1.7.3.2.1; 1.7.3.2.2; 1.7.3.2.3	5.9.2	120
24.5	Sample Matrix Controls	V1M2 Secs. 5.9.2; 5.9.3. V1M4 Secs. 1.7.3 ; 1.7.3.3; 1.7.3.3.1; 1.7.3.3.2; 1.7.3.3.3	5.9.2	121
24.6	Acceptance Criteria (Control Limits)	V1M2 Sec. 5.9.3. V1M4 Secs. 1.7.4.2; 1.7.4.3		122
24.7	Additional Procedures To Assure Quality Control	V1M2 Sec. 5.9.3. V1M4 Sec. 1.7.3.4		124
25.0	REPORTING RESULTS			125
25.1	Overview	V1M2 Secs. 5.10.1; 5.10.2; 5.10.8	5.10.1; 5.10.2; 5.10.8	125
25.2	Test Reports	V1M2 Secs. 5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8; 5.10.10; 5.10.11	5.10.1; 5.10.2; 5.10.3.1; 5.10.3.2; 5.10.5; 5.10.6; 5.10.7; 5.10.8	125
25.3	Reporting Level Or Report <u>Type</u>	V1M2 Secs. 5.10.1; 5.10.7; 5.10.8	5.10.1; 5.10.7; 5.10.8	127
25.4	Supplemental Information for Test	V1M2 Secs. 5.10.1; 5.10.3.1; 5.10.5	5.10.1; 5.10.3.1; 5.10.5	128
25.5	Environmental Testing Obtained From Subcontractors	V1M2 Secs. 4.5.5; 5.10.1; 5.10.6	5.10.1; 5.10.6	128
25.6	Client Confidentiality	V1M2 Secs. 4.1.5; 5.10.7	4.1.5; 5.10.7	128
25.7	Format Of Reports	V1M2 Sec. 5.10.8	5.10.8	129
25.8	Amendments To Test Reports	V1M2 Sec. 5.10.9	5.10.1; 5.10.9	129
25.9	Policies On Client Requests For Amendments	V1M2 Secs. 5.9.1; 5.10.9	5.9.1; 5.10.1; 5.10.5; 5.10.9	129

## LIST OF TABLES

Table No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005 (E) Reference	Page No.
14-1	Records Index		4.13.1.1	59
14-2	Special Record Retention Requirements			60
15-1	Types Of Internal Audits And Frequency		4.14.1	65
20-1	Instrumentation L		5.5.4; 5.5.5	101

## LIST OF FIGURES

Figure No.	Title	2009 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	Page No.
4-1	Corporate And Laboratory Organization Charts	V1M2 Sec. 4.1.5	4.1.3; 4.1.5; 4.2.6	22
12-1	Corrective Action Report	V1M2 Sec. 4.11.6. V1M4 Sec. 1.7.4.1	4.11.2	52
19-1	Demonstration Of Capability Documentation			91
19-2	Work Flow			92
23-1	Chain Of Custody (COC)			116
23-2	Sample Acceptance Policy	V1M2 Sec. 5.8.6; 5.8.7.1. V1M4 Sec. 1.7.5		117
23-3	Login Sample Receipt Checklist		5.8.3	118

## LIST OF APPENDICES

Appendix No.	Title	Page No.
1	Laboratory Floor Plan	131
2	<b>Glossary/Acronyms</b>	137
3	Laboratory Certifications, Accreditations, Validations	166

**REFERENCED CORPORATE SOPs AND POLICIES**

<b>SOP / Policy Reference</b>	<b>Title</b>
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CW-Q-S-003	Internal Auditing
CA-Q-S-006	Detection Limits
CW-Q-S-004	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOP)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-T-P-001	Qualified Products List
CW-F-S-007	Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual



## REFERENCED LABORATORY SOPs

SOP Reference	Title
SA-AN-041	Reagent and Standard Materials Procedures
SA-AN-100	Laboratory Support Equipment (Verification and Use)
SA-CU-001	Sample Receipt Procedures
SA-CU-015	Preparation of Sampling Kits
SA-EX-015	Toxicity Compound Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)
SA-EX-030	Liquid Extraction Procedures: Continuous Liquid-Liquid & Separatory Funnel
SA-EX-040	Soil Extraction Procedures: Microwave and Sonication
SA-EX-042	Waste Dilution Extraction
SA-FD-005	Field Sampling Procedures
SA-GE-001	Measurement of Analytes Using Konelab Autoanalyzer
SA-GE-010	Bomb Combustate Preparation
SA-GE-040	Cyanide: Total, Amenable, and Weak Acid Dissociable
SA-GE-085	Sulfide: Titrimetric Preparation and Analysis
SA-GE-113	Disinfection Byproduct Anions by Ion Chromatography
SA-GE-115	Anions by Ion Chromatography
SA-GE-132	Sulfite
SA-GE-133	Total Residual Chlorine by Iodometric Titration
SA-GE-140	Flashpoint and Ignitability
SA-GE-157	Oil & Grease and Petroleum Hydrocarbons by Gravimetry
SA-GE-160	Methylene Blue Active Substances (MBAS)
SA-GE-165	Total Recoverable Phenolics
SA-GE-187	Organic Halides: Adsorbable (AOX) & Total (TOX)
SA-GE-189	UV - Absorbing Organic Constituents (UV-254 & SUVA)
SA-GE-190	Solid / Residue Determinations
SA-GE-191	pH Determination
SA-GE-193	Measurement of Analytes using PC Titrate Analyzer
SA-GE-194	Dissolved Oxygen: Winkler Titration
SA-GE-195	Chemical Oxygen Demand: Colorimetric Method
SA-GE-196	Density and Specific Gravity
SA-GE-197	Color
SA-GE-198	Free Liquids by Paint Filter Liquids Test
SA-GE-201	Total Hardness as CaCO <sub>3</sub> by Titrimetric EDTA
SA-GE-202	Oxygen Demand: Biochemical Oxygen Demand (BOD), and Carbonaceous Biochemical Oxygen Demand (CBOD), and Nitrogenous Biochemical Oxygen Demand (NBOD)
SA-GE-204	Carbon Content in Water: Total Carbon (TC), Total Organic Carbon (TOC), and Total Inorganic Carbon
SA-GE-205	Odor
SA-GE-206	Turbidity
SA-GE-208	Nitrate and Nitrate Plus Nitrite: Lachat Procedure
SA-GE-210	Total Kjeldahl Nitrogen and Total Phosphorus via Lachat Autoanalyzer

SOP Reference	Title
SA-LC-070	Carbamate Pesticides by HPLC
SA-LC-071	Glyphosate by HPLC
SA-LC-072	Diquat and Paraquat by HPLC
SA-ME-021	Digestion Procedures for Solids for Hexavalent Chromium
SA-ME-028	Mercury: Preparation and Analysis
SA-ME-050	Digestion Procedures Liquids for ICP and ICP/MS
SA-ME-051	Digestion Procedures Solids for ICP and ICP/MS
SA-ME-070	Elements by ICP
SA-ME-074	Elements by ICP/MS
SA-PM-001	Project Management
SA-QA-001	Document Control Program
SA-QA-002	Data Generation and Review
SA-QA-005	Preventive and Corrective Action
SA-QA-006	Training Procedures
SA-QA-007	Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)
SA-QA-008	Evaluation of Chromatographic Data
SA-QA-010	Validation of New Analytical Capabilities and Instrumentation
SA-QA-015	Homogenization, Compositing, and Segregation of Samples
SA-QA-016	Evaluation of Calibration Curves
SA-QA-017	Analytical Batching and Evaluation of Batch QC Data
SA-SG-045	Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by GC/ECD
SA-SG-046	Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Drinking Water by GC/ECD
SA-SG-060	Microextractables by GC/ECD
SA-SG-062	Haloacetic Acids by Gas Chromatography
SA-SG-065	Chlorinated Herbicides by GC/ECD: Preparation and Analysis
SA-SG-070	Diesel Range Organics (DRO), Oil Range Organics (ORO), & Petroleum Product Identification by GC/FID
SA-SG-071	Dicofol and DCBP by GC/ECD
SA-SM-002	Semivolatile Organic Compounds in Drinking Water by GC/MS
SA-SM-007	Polychlorinated Biphenyls (PCBs) by GC/MS
SA-SM-030	Endothall by GC/MS
SA-SM-031	Chlorinated Phenolics by GC/MS
SA-SM-033	Semivolatile Compounds by GC/MS
SA-VO-001	Preparation, Screening, and Storage of Volatile Samples
SA-VO-002	Volatile Compounds in Drinking Water by GC/MS
SA-VO-003	Acetates in the Pharmaceutical Industry by GC/MS
SA-VO-004	Volatile Compounds by GC/MS
SA-VO-005	Gasoline Range Organics (GRO) by GC/FID
SA-VO-006	Solvents by Direct Aqueous Injection (DAI) Using GC/FID
SA-VO-007	Dissolved Gases in Water

## SECTION 3. INTRODUCTION, SCOPE, AND APPLICABILITY

### 3.1 Introduction and Compliance References

TestAmerica Savannah's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water*, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories*, EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Version 4.2, October 2010.
- U.S. Department of Defense (DoD)/Department of Energy (DOE) *Consolidated Quality Systems Manual (QSM) for Environmental Laboratories*, Version 5.0, July 2013,
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, *Standard Methods for the Examination of Water and Wastewater*, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup>, 21<sup>st</sup>, and on-line Editions.
- U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP)*, Version 5

### 3.2 Terms and Definitions

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control samples. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

### **3.3 Scope / Fields of Testing**

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge, and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in the Methods Listing housed in the laboratory's information management system (i.e., TALS). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### **3.4 Management of the Manual**

#### **3.4.1 Review Process**

The template on which this manual is based is reviewed annually by Corporate Quality Management personnel to assure that it remains in compliance with Section 3.1. This manual itself is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves

such changes according to our Document Control and Updating procedures (refer to SOP SA-QA-001).

## **SECTION 4. MANAGEMENT REQUIREMENTS**

### **4.1 Overview**

TestAmerica Savannah is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., Chief Executive Officer (CEO), Executive Vice President (VP) Operations, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate and TestAmerica Savannah is presented in Figure 4-1.

### **4.2 Roles and Responsibilities**

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### **4.2.1 Additional Requirements for Laboratories**

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Savannah laboratory.

##### **4.2.1.1 Quality Assurance (QA) Manager or Designee**

The QA Manager has responsibility and authority to ensure the continuous implementation and improvement of the quality system based on ISO/IEC 17025, DOD ELAP, and TNI. The QA Manager is independent of production; reports directly to the Laboratory Director; and has access to Corporate QA for advice and resources. The QA Manager is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. The QA Manager directs the activities of the QA Department to accomplish specific responsibilities, which include, but are not limited to:

- Serving as the focal point for QA/QC in the laboratory.
- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.



- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities. Ensuring all personnel understand their contributions to the Quality System.
- Having documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- Maintaining records of all ethics-related training, including the type and proof of attendance.
- Maintaining, improving, and evaluating the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitoring standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating the document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviewing a percentage of all final data reports for consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, format, holding time, sensibility, and completeness of the project file contents.
- Reviewing of external audit reports and data validation requests.
- Following-up with audits to ensure client QAPP requirements are met.
- Establishing of reporting schedule and preparation of various quality reports for the Laboratory Director, clients, and/or Corporate QA.
- Developing of suggestions and recommendations to improve quality systems.
- Researching current state and federal requirements and guidelines.
- Managing the QA team to enable communication and to distribute duties and responsibilities.
- Evaluating of the thoroughness and effectiveness of training.
- Ensuring compliance with ISO/IEC 17025, DOD ELAP, and TNI.

#### **4.2.1.2 Technical Manager/Director**

The Technical Manager is accountable for all analyses and analysts under their experienced supervision and for compliance with ISO 17025, DOD ELAP, and TNI. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods (i.e., SOPs) with regard to quality, integrity, regulatory requirements, and optimum and efficient production techniques; and subsequent analyst training and

interpretation of the SOPs for implementation and unusual project samples. He ensures that the SOPs are properly managed and adhered to at the bench.

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, and the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting project QAPPs, ensuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting, and preventive maintenance.
- Enhancing efficiency and improving quality through technical advances and improved TALS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from "cradle to grave," ensuring that no time is lost in locating samples.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.

#### **4.2.1.2      Laboratory Director**

Specific responsibilities include, but are not limited to, the following:

- Directs and provides guidance to Operations Manager and Project Managers
- Develops and maintains company-client relationships
- Reviews proposals
- Interviews and hires technical and administrative personnel
- Other administrative and budgetary functions

#### **4.2.1.3      Operations Manager**

Specific responsibilities include, but are not limited to, the following:

- Coordinates all production activities
- Works with Project Managers to ensure project objectives are met
- Provides guidance to Supervisors
- Interviews and hires laboratory personnel
- Establishes production priorities and coordinates day-to-day operation of the laboratory

#### **4.2.1.5 Compliance Officer/Environmental Health and Safety Manager**

Specific responsibilities include, but are not limited to, the following:

- Provides technical assistance in complying with corporate policies concerning safety, waste, and shipping
- Assists Laboratory Director, Operations Manager, and Project Managers in developing appropriate safety precautions for new projects
- Monitors collection and disposal of chemical wastes
- Ensures employees comply with safety and waste disposal plans

#### **4.2.1.6 Supervisor**

Specific responsibilities include, but are not limited to, the following:

- Organizes workflow in the department
- Assures adequate inventory of reagents and equipment
- Ensures effective maintenance and repair of instrumentation
- Investigates and evaluates new methodology and equipment
- Ensures proper training is conducted
- Reviews data, assures quality objectives are met for each project, and approves results

#### **4.2.1.7 Analyst/Chemist**

Specific responsibilities include, but are not limited to, the following:

- Performs preparation and/or analysis of samples using approved procedures
- Calculates, checks, and reports data in accordance with approved SOP and the Laboratory Quality Manual
- Performs instrument maintenance and maintains instrument logs
- Maintains proper documentation of all analytical steps

#### **4.2.1.8 Lab Technician**

Specific responsibilities include, but are not limited to, the following:

- Assists analysts in sample preparation and data collection
- Performs routine checks for data quality objectives - surrogate recoveries, LCS/MS recoveries, initial evaluation of dilutions, internal standards areas, and method blanks
- Assists analysts in maintaining traceability of standards and samples
- Assists analysts in preparing samples, extracts, or digests for analysis
- Checks samples for proper preservation and maintains department sample receipt and chain-of-custody logs

#### **4.2.1.9 Client Services Director**

Specific responsibilities include, but are not limited to, the following:

- Coordinates marketing efforts with General Manager, Laboratory Director, Project Managers, and laboratory marketing group
- Supervises Project Managers
- Coordinates proposal and contract review and response process
- Responds to client inquiries

#### **4.2.1.10 Project Manager**

Specific responsibilities include, but are not limited to, the following:

- Serves as primary contact with client on individual job tasks

- Prepares work plans; schedules manpower allocations
- Initiates all procurement for each project
- Provides day-to-day coordination of the project team
- Coordinates financial and contractual aspects of the projects
- Provides formatting and technical review of all reports
- Provides day-to-day communication with the client
- Exercises final review and approval on all reports and invoices for the project
- Responds to post project inquiries

#### 4.2.1.11 **Custody Supervisor**

Specific responsibilities include, but are not limited to, the following:

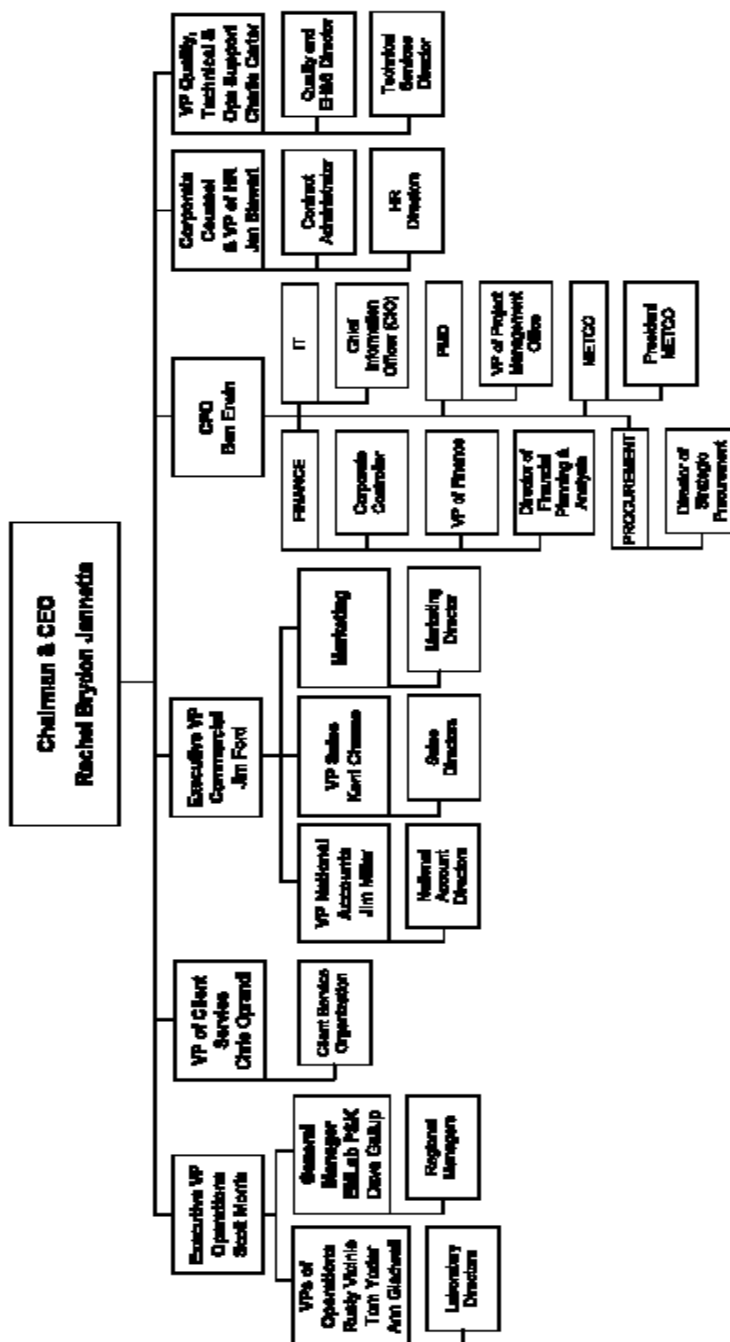
- Schedules bottle orders and supervises bottle prep staff
- Supervises sample custody staff
- Coordinates with Project Managers and Field/Sampling Supervisor on scheduling field sampling efforts
- Identifies and documents custody discrepancies and notifies Project Managers about custody problems

#### 4.3 **Deputies**

The following table defines who assumes the responsibilities of key personnel in their absence:

<b>Key Personnel</b>	<b>Deputy</b>
Laboratory Director	Client Services Director
QA Manager	Laboratory Director
Operations Manager	Laboratory Director
Technical Director/Manager	Laboratory Director
EHS Coordinator	QA Manager

Figure 4-1. Corporate and Laboratory Organization Charts



May 2014

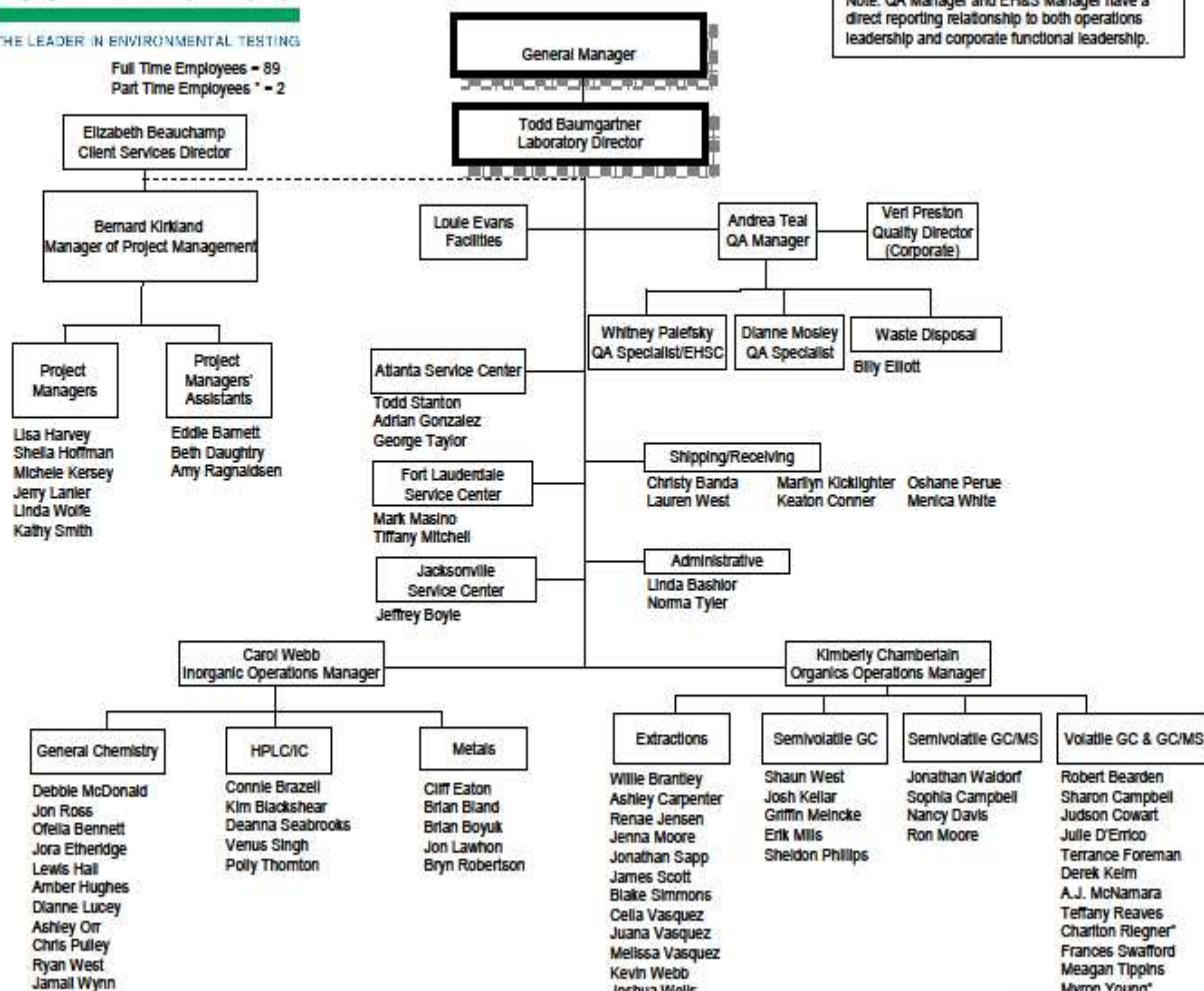


THE LEADER IN ENVIRONMENTAL TESTING

Full Time Employees = 89  
Part Time Employees = 2

## Savannah Laboratory Organization

Note: QA Manager and EH&S Manager have a direct reporting relationship to both operations leadership and corporate functional leadership.



Revised 05.14.14



## **SECTION 5. QUALITY SYSTEM**

### **5.1 Quality Policy Statement**

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ To comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard, and the DOD QSM, and to continually improve the effectiveness of the quality management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

### **5.2 Ethics and Data Integrity**

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002)
- Procedures and guidance for recalling data if necessary. (Corporate SOP No. CW-L-S-002)
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Production of results, which are accurate and include QA/QC information that meet client pre-defined Data Quality Objectives (DQOs).

- Presentation of services in a confidential, honest, and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

### **5.3 Quality System Documentation**

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- Corporate SOPs and Policies – Corporate SOPs and Policies are developed for use by all laboratories. The policies described therein are typically incorporated into laboratory-specific SOPs, or the Corporate documents may be incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs – General and Technical

#### **5.3.1 Order of Precedence**

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

### **5.4 QA/QC Objectives for the Measurement of Data**

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term “*analytical quality control*”. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPPs) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

#### **5.4.1      Precision**

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

#### **5.4.2      Accuracy**

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or matrix spikes (MS). A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

#### **5.4.3      Representativeness**

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be

documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

#### **5.4.4 Comparability**

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

#### **5.4.5 Completeness**

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

#### **5.4.6 Selectivity**

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc.

#### **5.4.7 Sensitivity**

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (e.g., Method Detection Limit) or quantified (e.g., Reporting Limit).

#### **5.5 Criteria for Quality Indicators**

The laboratory maintains Method Limit Groups in TALS that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date,

is updated each time new limits are generated, and are managed by the laboratory's QA Department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP SA-QA-017: *Evaluation of Batch QC Data*.

## **5.6 Statistical Quality Control**

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory that are entered into the Laboratory Information Management System (i.e. TALS). The Quality Assurance Department maintains an archive of all limits used within the laboratory and stores these values in TALS. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the laboratory develops such limits from recent data in the QC database of the TALS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the TALS analyte database. As sample results and the related QC are entered into TALS, the sample QC values are compared with the limits in TALS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

### **5.6.1 QC Charts**

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (recommended annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*. The QA Manager evaluates control charts to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

## **5.7 Quality System Metrics**

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

## **SECTION 6. DOCUMENT CONTROL**

### **6.1 Overview**

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOPs)
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers, and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP SA-QA-001: *Document Control Program*.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

### **6.2 Document Approval and Issue**

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, an employee submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.



The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

### **6.3        Procedures for Document Control Policy**

For changes to the QA Manual, refer to SOP refer to SOP SA-QA-001: *Document Control Program*.

Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA Department. Electronic controlled copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP SA-QA-001: *Document Control Program*.

Electronic copies of current documents (including QA Manuals, SOPs, Forms, Work Instructions, etc.) are maintained by the QA Department and distributed electronically via the QA Navigator.

### **6.4        Obsolete Documents**

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, for hardcopy distribution, obsolete documents are collected from employees according to distribution lists and are destroyed. At least one copy of the obsolete document is archived according to SOP SA-QA-001: *Document Control Program*.

## **SECTION 7. SERVICE TO THE CLIENT**

### **7.1 Overview**

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the laboratory's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals, and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel, and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the laboratory to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## **7.2 Review Sequence and Key Personnel**

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements, and that the lab has the capacity to meet the clients turn around needs.

For new, complex or large projects, the proposed contract is given to the Sale Directors, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002: Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above. The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the designated personnel is not available to review the contract, his back-up will fulfill the review requirements.

The Contracts Director and the local Proposal Coordinator maintain copies of all signed contracts.

## **7.3 Documentation**

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained by the Proposal Coordinator.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract.

### **7.3.1 Project-Specific Quality Planning**

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before

and during the project. QA Department involvement may be needed to assist in the evaluation of custom QC requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PMs do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each Project in TALS as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, email, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the supervisor.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

#### **7.4      Special Services**

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

**Note:** ISO/IEC 17025 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.

- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

#### **7.5      Client Communication**

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers, Operations Managers, Supervisors, and the QA Manager are available to discuss any technical questions or concerns that the client may have.

#### **7.6      Reporting**

The laboratory works with our clients to produce any special communication reports required by the contract.

#### **7.7      Client Surveys**

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica's Sales and Marketing teams periodically develop laboratory and client specific surveys to assess client satisfaction.

## SECTION 8. SUBCONTRACTING OF TESTS

### 8.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica’s Corporate SOPs on Subcontracting Procedures (CA-L-S-002).

When outsourcing analytical services, the laboratory will ensure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI and ISO/IEC 17025, and/or the client’s Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client’s analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

Project Managers (PMs), Client Service Managers (CSM), or Account Executives (AE) for the export lab (i.e., the TestAmerica laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing, and, when possible, approval from the client shall be retained in the project folder. Standard TestAmerica Terms & Conditions include the flexibility to subcontract samples within the TestAmerica laboratories. Therefore, additional advance notification to clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

**Note:** In addition to the client, some regulating agencies (e.g., USDA) or contracts (e.g., certain USACE projects) may require notification prior to placing such work.

### 8.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task. Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an email from the client in the project folder.



- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract laboratory. Verify necessary accreditation, where applicable, (e.g., on the subcontractor's TNI, A2LA accreditation, or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned, and/or minority-owned businesses;
- TNI or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for worksharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an email is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an email is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to the Corporate Quality Information Manager (QIM) for review. Once all documents are reviewed for completeness, the Corporate QIM will forward the documents to the Purchasing Manager for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site.

Complaints are posted using the Vendor Performance Report.

- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers, and Sales Personnel.

### **8.3 Oversight and Reporting**

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and Corporate Counsel can tailor the document or assist with negotiations, if needed. The PM responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples subcontracted within TestAmerica. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which laboratory produced the data for which methods and samples.

**Note:** The results submitted by a TestAmerica worksharing laboratory may be transferred electronically and the results reported by the TestAmerica worksharing laboratory are identified

on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

#### **8.4      Contingency Planning**

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this time. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

## **SECTION 9. PURCHASING SERVICES AND SUPPLIES**

### **9.1 Overview**

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Capital Expenditure, Controlled Purchase Requests and Fixed Asset Capitalization SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Company-Wide Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFPs allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards, and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

### **9.2 Glassware**

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

### **9.3 Reagents, Standards & Supplies**

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

#### **9.3.1 Purchasing**

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

#### **9.3.2 Receiving**

It is the responsibility of the Shipping and Receiving Department to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials are received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Safety Data Sheets (SDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

### 9.3.3 Specifications

Methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration date noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and Technical Manager must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified “clean” by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots may be verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer’s certification and traceability statements are maintained electronically.

#### **9.3.4      Storage**

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

#### **9.4            Purchase of Equipment / Instruments / Software**

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica’s Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed, and Purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and it is added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate for the specific intended application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs) if a new method, and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the TALS Administrator. The manufacturer’s operation manual is retained electronically.

#### **9.5            Services**

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

#### **9.6            Suppliers**



TestAmerica selects vendors through a competitive proposal/bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts, or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc. As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors.

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

#### **9.6.1      New Vendor Procedure**

TestAmerica employees who wish to request the addition of a new vendor must complete a JD Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or Technical Management are consulted with vendor and product selection that have an impact on quality.

## **SECTION 10. COMPLAINTS**

### **10.1 Overview**

The laboratory considers an effective client complaint handling process to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations, and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing, and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints, or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SA-QA-005: *Preventive and Corrective Action*.

### **10.2 External Complaints**

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOP-SA-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

### **10.3      Internal Complaints**

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing, and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

### **10.4      Management Review**

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly Report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

## **SECTION 11. CONTROL OF NON-CONFORMING WORK**

### **11.1 Overview**

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies, and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Supervisor for resolution. The Supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client requests that a special procedure be applied to a sample that is not standard laboratory practice. Based on a technical evaluation, the laboratory may accept or reject the request based on technical or ethical merit. Such a request would need to be approved by laboratory management and documented in the project files. Deviations to standard operating procedures must be noted in the final report.

### **11.2 Responsibilities and Authorities**

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CW-L-S-002), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to a member of Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Operations Manager, QA

Manager, and the Technical Manager. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, Executive VP of Operations, VP of Operations, and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

### **11.3      Evaluation of Significance and Actions Taken**

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

### **11.4      Prevention of NonConforming Work**

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

### **11.5      Method Suspension / Restriction (Stop Work Procedures)**

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required, and will be concluded with a discussion of the

steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target, or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be provided by the laboratory to the appropriate member of Corporate QA, which serves as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing, or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will not generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, or determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval may be given by final signature on the completed corrective action report.



## **SECTION 12. CORRECTIVE ACTION**

### **12.1 Overview**

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using NonConformance Memos (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1).

### **12.2 General**

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

**12.2.1 Non-Conformance Memo (NCM)** - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Discrepancies in materials / goods received vs. manufacturer packing slips.

**12.2.2 Corrective Action Report (CAR)** - is used to document the following types of corrective actions:

- Questionable trends that are found in the review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Client complaints
- Data recall investigations
- Identified poor process or method performance trends

- Excessive revised reports

This will provide background documentation to enable root cause analysis and preventive action.

### **12.3 Closed Loop Corrective Action Process**

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

#### **12.3.1 Cause Analysis**

- Upon discovery of a event requiring action, the event must be defined and documented. A CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

#### **12.3.2 Selection and Implementation of Corrective Actions**

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR is used for this documentation.

#### **12.3.3 Root Cause Analysis**

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with the problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

#### **12.3.4 Monitoring of the Corrective Actions**

- The Technical Manager, Operations Manager, and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes.
- The QA Manager reviews monthly NCRs and CARs for trends. Highlights are included in the QA Monthly Report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

#### **12.3.5 Follow-up Audits**

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

#### **12.4 Technical Corrective Actions**

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, SOP SA-QA-017: *Evaluation of Batch QC Data* and the analytical SOPs.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA Monthly Report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

## **12.5      Basic Corrections**

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

**Figure 12-1.**  
**Corrective Action Report**

<b>Section 1</b> <b><u>Summary of Problem / Finding</u></b>
Finding #:  Summary:  Date Due to Agency:
<b>Section 2</b> <b><u>Initial Investigation Summary</u></b>
Investigation Question #1: <ul style="list-style-type: none"><li>Is this issue chronic (i.e., were multiple instances cited, or is the potential for similar issues present), or acute (i.e., an isolated, anomalous, or non-routine occurrence)?</li></ul> Response:
Investigation Question #2: <ul style="list-style-type: none"><li>Are other departments likely to be impacted?</li></ul> Response:
Investigation Question #3: <ul style="list-style-type: none"><li>Can the root cause be readily established/addressed and action items identified without further inquiry, or is further action needed to perform a formal RCA Investigation and/or develop the Corrective Action Plan?</li></ul> <p>Note: If the root cause can be readily established/addressed and action items identified without further inquiry, then Section 3 does not need to be completed provided additional details are included in response to Investigation Question #4, below.</p> Response:
Investigation Question #4: <ul style="list-style-type: none"><li>Are there any additional comments worth noting? If so, please include.</li></ul> Response:
<b>Section 3</b> <b><u>Root Cause Analysis Summary</u></b>
RCA Investigation Lead:  RCA Investigation Team Members, if applicable:

RCA Question #1:

- Why was this finding cited?

Options:

1. Procedure/policy does not exist, is not adequate, or is not accurate.
2. Procedure/policy is in place, adequate, and accurate; however, employee did not comply.
3. Other

Response:

RCA Question #2:

- What are some underlying causes for the conclusion drawn in RCA Question #1 (i.e., what are some Quality System weaknesses indicated by this issue that also need to be addressed)?

Note: There may be more than one underlying cause/weakness, and each underlying cause/weakness may in turn have other underlying causes/weaknesses.

Examples:

1. Insufficient or incomplete method validation procedures.
2. Trend analysis was not performed or is insufficient.
3. Insufficient or incorrect detail in SOPs; SOPs out of date; SOPs do not match current practice, etc.
4. Missing or inadequate mechanism to capture information (e.g., form, spreadsheet, Data Types, etc.).
5. Missing or inadequate training.
6. Insufficient employee oversight / supervision.
7. Ineffective primary data review process.
8. Ineffective self-monitoring process (e.g., notebook review, secondary data review, internal audits, etc.).
9. Personnel problem, insufficient resources, lack of attention to detail, etc.
10. Insufficient reagent traceability or control procedures.
11. Poor communication channels.
12. Improper or inadequate equipment maintenance procedures.
13. Ineffective Document Control mechanisms
14. Ineffective sample scheduling mechanisms, workflow, backlogs, etc.
15. Other

Response:

RCA Question #3:

- Is a Data Recall, an SOP revision, or additional training needed?

Response:

RCA Question #4:

- Are there any additional comments worth noting? If so, please include.

Response:

#### **Section 4** **Corrective Action Assignments**

<<Based on the Initial Investigation and/or Root Cause Analysis Summary outlined above, what action items are needed to: 1) correct the original finding, and 2) minimize its recurrence? >>



Action Item #1:

Assigned Party:  
Due Date:  
Status:

Actions Taken:

Supporting Documentation Attached:

**Section 5**  
**Audit Response Documentation**

Laboratory Response sent to agency on: XXXXX, attached here.

**Section 6**  
**Subsequent Information / Documentation Requests from Agency**

Summary:

Assigned to:  
Due Date:

Documentation attached here:

**Section 7**  
**Additional Close-Out / Follow-Up and Comments**

A)  
This finding pertains to an isolated and/or anomalous event. The corrective action taken is sufficient to address this issue. No further action or follow-up is needed at this time to close out this item.  
Initial / Date:

B)  
An additional routine follow-up assessment is required to evaluate the effectiveness of the corrective action taken.

Follow-up Assigned To:

Due Date:

Documentation Needed:

Items used to assess effectiveness/sustainability of corrective action:

<<Include AD batch numbers, attach example logbook pages, etc., as applicable.>>

Choose One:

a) Corrective action has been implemented and is effective.

b) Similar problems have been noted. The corrective action has not been effective. Additional action is required.

Initial / Date:

**Table 12-1. General Corrective Action Procedures**

<b>QC Activity</b> <i>(Individual Responsible for Initiation/Assessment)</i>	<b>Acceptance Criteria</b>	<b>Recommended Corrective Action</b>
Instrument Blank <i>(Analyst)</i>	- Criteria in analytical SOP	- Prepare and analyze another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards <i>(Analyst)</i>	- Criteria in analytical SOP	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.
Initial Calibration Verification (Second Source ICV) <i>(Analyst)</i>	- Criteria within analytical SOP	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Verification (CCV) <i>(Analyst)</i>	- Criteria within analytical SOP	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) <i>(Analyst)</i>	- Criteria in TALS MLGs	- If matrix interferences are present, evaluate the LCS. - If the LCS is within acceptable limits the batch is acceptable.
Laboratory Control Sample (LCS) <i>(Analyst)</i>	- Criteria in TALS MLGs and SOP SA-QA-017	- Reanalyze LCS. - Batch must be re-prepared and/or re-analyzed.
Surrogates <i>(Analyst)</i>	- Criteria in TALS MLGs	- Individual sample must be repeated, unless obvious matrix interference is noted.
Method Blank <i>(Analyst)</i>	<1/2RL	- Reanalyze blank. - Determine source of contamination. - Re-prepare/re-analyze batch.

## **SECTION 13. PREVENTIVE ACTION / IMPROVEMENT**

### **13.1 Overview**

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems, and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results and evaluation of proficiency testing (PT) performance, data analysis and review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system:

- Identification of an opportunity for preventive action.
- Process for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

**13.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

## **SECTION 14. CONTROL OF RECORDS**

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

### **14.1 Overview**

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance, and disposal of quality and technical records. A record index is listed in Table 14-1. Technical records are maintained by the laboratory departments in the Data Archival folder on the Public\_QA Drive and are backed up as part of the regular network backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer- or hand-generated (some records may be in both formats).

**Table 14-1. Records Index<sup>1</sup>**

	<b>Record Types <sup>1</sup>:</b>	<b>Retention Time:</b>
<b>Technical Records</b>	<ul style="list-style-type: none"> <li>- Raw Data</li> <li>- Logbooks<sup>2</sup></li> <li>- Standards</li> <li>- Certificates</li> <li>- Analytical Records</li> <li>- MDLs/IDLs/DOCs</li> <li>- Lab Reports</li> </ul>	5 Years from analytical report issue*
<b>Official Documents</b>	<ul style="list-style-type: none"> <li>- Quality Assurance Manual (QAM)</li> <li>- Work Instructions</li> <li>- Policies</li> <li>- SOPs</li> </ul>	5 Years from document retirement date*
<b>QA Records</b>	<ul style="list-style-type: none"> <li>- Internal &amp; External Audits/Responses</li> <li>- Certifications</li> <li>- Corrective/Preventive Actions</li> <li>- Management Reviews</li> <li>- Method &amp; Software Validation / Verification Data</li> <li>- Data Investigation</li> </ul>	5 Years from archival*  <b>Data Investigation:</b> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
<b>Project Records</b>	<ul style="list-style-type: none"> <li>- Sample Receipt &amp; COC Documentation</li> <li>- Contracts and Amendments</li> <li>- Correspondence</li> <li>- QAPP</li> <li>- SAP</li> <li>- Telephone Logbooks</li> <li>- Lab Reports</li> </ul>	5 Years from analytical report issue*
<b>Administrative Records</b>	Finance and Accounting	10 years
	EH&S Manual and Permits	5 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies Technical Training Records	Refer to HR Manual

<sup>1</sup> Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance Log, Instrument Run Log, Preparation Logs (standard and samples), Standard and Reagent Receipt Logs, Balance Calibrations, Temperature Logs, etc.

\* Exceptions listed in Table 14-2.



**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

#### **14.1.2 Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

**Table 14-2. Special Record Retention Requirements**

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**14.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information. Electronic records are maintained in the Data Archival Folder on the Public\_QA drive, or in another applicable drive (such as Q-drive or I-drive). Refer to SOP SA-QA-001: *Document Control Program* for specific information on the archival, storage, and back-up of records.

**14.1.4** The recordkeeping system allows for historical reconstruction of all laboratory activities that produced the analytical data as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The chain of custody would indicate the name of the sampler.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set, etc. as per SOP SA-QA-001: *Document Control Program*. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument. Where an analysis is performed without an instrument, TALS sheets, bound logbooks, bench sheets, or spreadsheets are used to record and file data. Standard and reagent information is recorded in the TALS for each method.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

## **14.2      Technical and Analytical Records**

**14.2.1**      The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records, and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for performance of each analysis and reviewing results.

**14.2.2**      Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**14.2.3**      Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in TALS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are

indicated both in the TALS and on specific analytical report formats.

**14.2.4** All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a documented supervisory or peer review on a monthly basis.

### **14.3      Laboratory Support Activities**

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

#### **14.3.1      Sample Handling Records**

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

### **14.4      Administrative Records**

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

## **14.5 Records Management, Storage and Disposal**

All records (including those pertaining to test equipment), certificates, and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the TALS. Records are considered archived when noted as such in the records management system.

### **14.5.1 Transfer of Ownership**

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

### **14.5.2 Records Disposal**

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

## SECTION 15. AUDITS

### 15.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

**Table 15-1. Types of Internal Audits and Frequency**

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
QA Technical Audits	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee  (Refer to SOP No. CW-Q-S-003)	Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint Responsibility: a) QA Manager or designee, b) Technical Manager or designee  (Refer to SOP No. CW-Q-S-003)	SOP Compliance Review Frequency: - Every 2 years (non-DOD SOPs) - 100% of SOPs annually (DOD SOPs)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI field of testing or as dictated by regulatory requirements

#### 15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, TestAmerica's Data Integrity and Ethics Policies, TNI quality systems, client and state



requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness and sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

#### **15.1.2     QA Technical Audits**

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

#### **15.1.3     SOP Method Compliance**

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every year for DOD methods, or every two years for non-DOD methods. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities (new IDOC), reviews of the analyst work products will be performed.

#### **15.1.4     Special Audits**

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

#### **15.1.5     Performance Testing**

Single blind performance audits are employed for several reasons. One purpose is to provide corrective action for parameters judged to be unacceptable on external or internal performance audits. Periodic internal performance audits are also used to test parameters that are not routinely tested by external performance audits. Finally, single blind performance audits are employed to satisfy certain certification requirements, to satisfy auditors' specific requests for performance audit samples, or to provide additional evidence of data quality to clients with specific questions regarding laboratory performance.

The laboratory participates semi-annually in performance audits conducted through the analysis of Proficiency Testing (PT) samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-Potable Water, and Soil.

These PT studies are performed approximately six months apart. The first study of the year is usually performed in January, and the second study is usually performed in July. The PT results are submitted to certification agencies directly from the PT Provider. Remedial PT studies can be performed, as required, for any analytes scored as unacceptable. Root cause investigation into any unacceptable results must be initiated. Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

## **15.2      External Audits**

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

### **15.2.1      Confidential Business Information (CBI) Considerations**

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

## **15.3      Audit Findings**

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that

could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Operations and/or Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the laboratory's corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

## **SECTION 16. MANAGEMENT REVIEWS**

### **16.1 Quality Assurance Report**

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Quality Director, and the VP of Operations. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, VP of Operations, or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and VPs of Operations.

### **16.2 Annual Management Review**

The senior laboratory management team (Laboratory Director, Operations Manager, QA Manager) conducts a review annually of its quality systems and TALS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives, and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The TALS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the TALS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CW-Q-S-004 & Work Instruction No. CW-Q-WI-003) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
- Adequacy of staff, equipment and facility resources.

- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

### **16.3 Potential Integrity Related Managerial Reviews**

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, Executive VP of Operations, VP of Client & Technical Services, VPs of Operations, and Quality Directors receive a monthly report from the Corporate Quality and EHS Director summarizing any current data integrity or data recall investigations. The VPs of Operations are also made aware of progress on these issues for their specific labs.

## **SECTION 17. PERSONNEL**

### **17.1 Overview**

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures, and records management.

Laboratory management is responsible for formulating goals for laboratory staff with respect to education, training, and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

### **17.2 Education and Experience Requirements for Technical Personnel**

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform, or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page. (Also see Section 4 for position descriptions/responsibilities).



Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	Or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – <b><u>General</u></b>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry  An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director – <b><u>Wet Chem</u></b> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Director - Microbiology	<p>Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology</p> <p>An advanced (MS, PhD.) degree may substitute for one year of experience</p>	And 2 years of relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics (New Hires)	1 week of hire	All
Ethics (Comprehensive)	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics (Comprehensive Refresher)	Annually	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood, and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques, or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analyst's knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicating to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the SOP SA-QA-006: *Training Procedures*.

#### **17.4 Data Integrity and Ethics Training Program**

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive ethics training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

## **SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS**

### **18.1 Overview**

The laboratory is a 55,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

### **18.2 Environment**

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control, and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and TALS are regulated to protect against raw data loss.

### **18.3      Work Areas**

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas available to ensure unencumbered work. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

### **18.4      Floor Plan**

A floor plan can be found in Appendix 1.

### **18.5      Building Security**

Building keys and alarm codes are distributed to employees as necessary.

Employees wear photographic identification name cards while on the premises.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed. Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.



## **SECTION 19. TEST METHODS AND METHOD VALIDATION**

### **19.1 Overview**

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage, and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

### **19.2 Standard Operating Procedures (SOP)**

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints, as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP SA-QA-001: *Document Control*.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

### **19.3 Laboratory Methods Manual**

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

## 19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### 19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup>/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.

- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### **19.4.2     Demonstration of Capability**

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method, or personnel (e.g., analyst hasn't performed the test within the last 12 months).

**Note:** The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve, and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the laboratory informs the client of its procedure for working with unusual compounds.

#### **19.4.3 Initial Demonstration of Capability (IDOC) Procedures**

Refer to SOP SA-QA-006: *Training Procedures* for information on performing Initial Demonstrations of Capability (IDOC).

A certification statement (refer to Figure 19-1) can be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

#### **19.5 Laboratory Developed Methods and Non-Standard Methods**

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

#### **19.6 Validation of Methods**

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to

meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

#### **19.6.1 Method Validation and Verification Activities for All New Methods**

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

##### **19.6.1.1 Determination of Method Selectivity**

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

##### **19.6.1.2 Determination of Method Sensitivity**

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

##### **19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)**

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

##### **19.6.1.4 Determination of Interferences**

A determination that the method is free from interferences in a blank matrix is performed.

##### **19.6.1.5 Determination of Range**

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### **19.6.1.6 Determination of Accuracy and Precision**

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### **19.6.1.7 Documentation of Method**

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### **19.6.1.8 Continued Demonstration of Method Performance**

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

#### **19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)**

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements. Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)* for details on the laboratory's MDL process.

#### **19.8 Instrument Detection Limits (IDL)**

The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of instrument blanks and calculating 3 x the absolute value of the standard deviation.



If IDL is > than the MDL, it may be used as the reported MDL.

### **19.9      Verification of Detection and Reporting Limits**

Once the MDL is determined, it must be verified on each instrument used for the given method. TestAmerica defines the DoD QSM Detection Limit (DL) as being equal to the MDL. TestAmerica also defines the DoD QSM Limit of Detection (LOD) as being equal to the lowest concentration standard that successfully verifies the MDL, also referred to as the MDLV standard. MDL and MDLV standards are extracted/digested and analyzed through the entire analytical process. The MDL and MDLV determinations do not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDLV standard is not successful, then the laboratory will redevelop their MDL or perform and pass two consecutive MDLVs at a higher concentration and set the LOD at the higher concentration. Initial and quarterly verification is required for all methods listed in the laboratory's DoD ELAP Scope of Accreditation. Refer to the laboratory SOP SA-QA-008 for further details.

The laboratory quantitation limit is equivalent to the DoD Limit of Quantitation (LOQ), which is at a concentration equal to or greater than the lowest non-zero calibration standard. The DoD QSM requires the laboratory to perform an initial characterization of the bias and precision at the LOQ and quarterly LOQ verifications thereafter. If the quarterly verification results are not consistent with three-standard deviation confidence limits established initially, then the bias and precision will be reevaluated and clients contacted for any on-going projects. For DoD projects, TestAmerica makes a distinction between the Reporting Limit (RL) and the LOQ. The RL is a level at or above the LOQ that is used for specific project reporting purposes, as agreed to between the laboratory and the client. The RL cannot be lower than the LOQ concentration, but may be higher.

### **19.10      Retention Time Windows**

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

### **19.11      Evaluation of Selectivity**

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, and chromatography retention time windows.

### **19.12      Estimation of Uncertainty of Measurement**

**19.12.1** Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty” (i.e., the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor  $k=2$ ).

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of  $k = 3$ . As an example, for a reported result of 1.0mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5mg/L.

Refer to SOP SA-QA-017: *Evaluation of Batch QC Data* for more information on this topic.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., EPA 524.2, EPA 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

### **19.13 Sample Reanalysis Guidelines**

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as ‘reanalysis’) may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will

reanalyze samples at a client's request with the following caveats. Client-specific, contractual Terms and Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within  $\pm 1$  reporting limit for samples  $\leq 5x$  the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the laboratory was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to non-homogenous samples, Encores/Terracores, and sodium bisulfate preserved samples.

#### **19.14 Control of Data**

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### **19.15 Computer and Electronic Data Related Requirements**

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the TestAmerica LIMS System (TALS) which is a custom in-house developed TALS system that has been highly customized to meet the needs of the laboratory. It is referred to as TALS for the remainder of this section. The TALS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.15.5.1 Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal TALS permissions procedure.

- TALS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails, and controlled access.

**19.15.5.2 Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure

storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

**19.15.5.3 Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

**19.15.6 Data Reduction**

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to approving the data in TALS.

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices* and SOP SA-QA-008: *Evaluation of Chromatographic Data*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

**19.15.6.1** All raw data is retained in the laboratory benchsheets, computer file (if appropriate), and/or runlog. All criteria pertinent to the method are recorded. The documentation is recorded at the time observations or calculations are made and each person involved is readily identified.

**19.15.6.2** In general, concentration results are reported in milligrams per liter (mg/L) or micrograms per liter ( $\mu\text{g/L}$ ) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ( $\mu\text{g/kg}$ ) for solids. For values greater than 10,000 mg/L, results can be reported in percent, i.e., 10,000 mg/L = 1%. Units are defined in each laboratory SOP.

**19.15.6.3** In general, results are reported to 2 significant figures on the final report.

**19.15.6.4** For those methods that do not have an instrument printout or an instrumental output compatible with the TALS, the raw results and dilution factors are entered directly into TALS by the analyst, and the software calculates the final result for the analytical report.

**19.15.6.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the TALS, the raw results and dilution factors are transferred into TALS electronically. Electronic data from instruments are saved electronically in a daily

folder on the system (Target or instrument computer). For instruments that print out calibrations and concentrations, the data are retained with the data file. The data file is stored in the Archival Folder on the Public\_QA. Periodically, these files are transferred to the server and, eventually, to a tape file.

#### **19.15.7 Logbook / Worksheet Use Guidelines**

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

**19.15.7.1** Corrections are made following the procedures outlined in Section 12.

**19.15.7.2** Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.

**19.15.7.3** Unused portions of pages must be "Z"ed out, signed and dated.

**19.15.7.4** Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Department controls all worksheets following the procedures in Section 6.

#### **19.15.8 Review / Verification Procedures**

Data review procedures are outlined in the analytical SOPs and SOP SA-QA-002: *Data Generation and Review* and ensure that data reported are free from calculation and transcription errors and that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing manual integrations to ensure the authenticity of the data (SOP SA-QA-008). The general review concepts are discussed below; more specific information can be found in the SOPs.

**19.15.8.1** The data review process at TestAmerica Savannah starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information into the TALS. The Project Management Assistant reviews the transaction of the chain-of-custody forms and inputs the required analyses. The Project Managers perform final review of the chain-of-custody forms and entered information.

**19.15.8.2** The next level of data review occurs with the analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements. The analysts transfer the data into the TALS. To ensure data compliance, another analyst/supervisor performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blanks, initial and continuing calibrations, laboratory control samples, sample data, qualifiers, manual integrations, and spike information are evaluated. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed

- Samples have unusually high results
- Samples exceed a known regulatory limit
- Raw data indicates some type of contamination or poor technique
- Inconsistent peak integration is observed
- Transcription errors are identified
- Results are outside of calibration range

**19.15.8.3** Unacceptable analytical results may require reanalysis of the samples. Problems may be brought to the attention of the Laboratory Director, Project Manager, Operations Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation, if needed. Corrective action is initiated whenever necessary.

**19.15.8.4** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/narratives are present, data qualifiers are appropriate, and project-specific requirements are met. The following are some examples of chemical relationships that can be reviewed (if data is available):

- Total Results are  $\geq$  Dissolved results (e.g. metals)
- Total Solids (TS)  $\geq$  Total Dissolved Solids (TDS) or Total Suspended Solids (TSS)
- TKN  $\geq$  Ammonia
- Total Phosphorus  $\geq$  Orthophosphate
- COD  $\geq$  TOC
- Total Cyanide  $\geq$  Amenable Cyanide
- TDS  $\geq$  individual anions

**19.15.8.5** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and sends to the client.

**19.15.8.6** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

#### **19.15.9      Manual Integrations**

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline for our internal SOP No. SA-QA-008, entitled *Evaluation of Chromatographic Data*.



- 19.15.9.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- 19.15.9.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 19.15.9.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- 19.15.9.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

**Figure 19-1. Demonstration of Capability Documentation**

**TRAINING DOCUMENTATION FORM  
DEMONSTRATION OF CAPABILITY**

Laboratory Name: TestAmerica Savannah  
Address: 5102 LaRoche Avenue  
Savannah, GA 31404

Date Completed: \_\_\_\_\_

Analyst Name: \_\_\_\_\_

Prep Analyst Name (s): \_\_\_\_\_

Analytical Test Method: \_\_\_\_\_

Prep Method: \_\_\_\_\_

Matrix: ☐ Soil ☐ Aqueous ☐ Other

Analytical SOP Document Control Number: \_\_\_\_\_

Prep SOP Document Control Number: \_\_\_\_\_

Analyte, Class of Analytes, or Measured Parameters: \_\_\_\_\_


If PT Study is used as DOC, list the PT Number: \_\_\_\_\_

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program and/or other state and federal programs have completed the Demonstration of Capability.
2. The test method(s) was performed by the analyst(s) identified on this certification.
3. A copy of test method(s) and laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

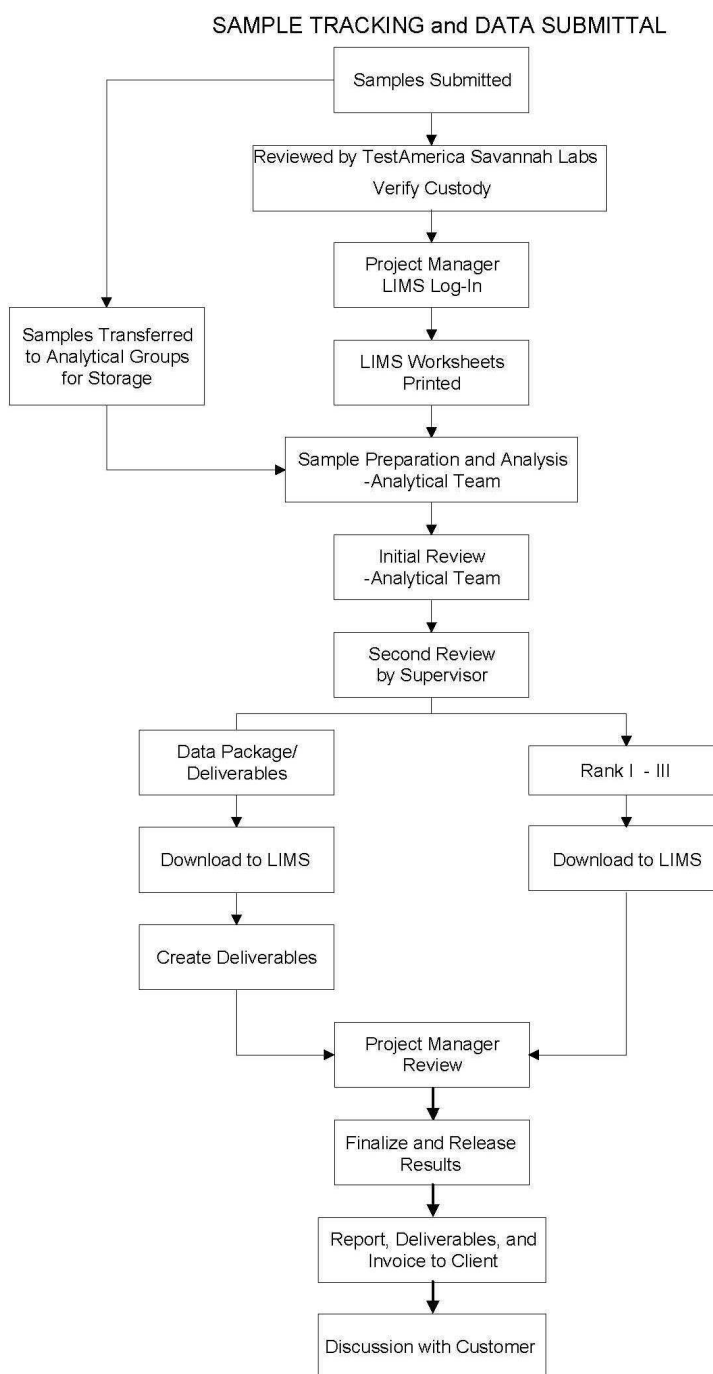
_____ Technical Director's Name	_____ Signature	_____ Date
_____ Quality Assurance Officer's Name	_____ Signature	_____ Date

FQA049:08.13.07.6



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**Figure 19-2. Work Flow**



## **SECTION 20. EQUIPMENT AND CALIBRATIONS**

### **20.1 Overview**

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

### **20.2 Preventive Maintenance**

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control (e.g. CCV run on 'date' was acceptable, or

instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed.

If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

## **20.3      Support Equipment**

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

### **20.3.1      Weights and Balances**

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage, or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

### **20.3.2     pH, Conductivity, and Turbidity Meters**

The pH meters used in the laboratory are accurate to  $\pm 0.1$  pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult the analytical SOPs for further information.

### **20.3.3     Thermometers**

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer at temperatures bracketing the range of use. IR thermometers, digital probes, and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4 degrees) and frozen (0 to -5 degrees), per the Drinking Water Manual.

The mercury NIST thermometer is recalibrated every three years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented electronically. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in equipment-specific logbooks or TALS sample batches. More information on this subject can be found in SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.

### **20.3.4     Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators**

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day – including weekends and holidays (i.e., 7 days a week).

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.



Sample storage refrigerator temperatures are kept between  $> 0^{\circ}\text{C}$  and  $\leq 6^{\circ}\text{C}$ .

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and procedure-specific logbooks.

### **20.3.5 Autopipettors, Dilutors, and Syringes**

Mechanical volumetric dispensing devices including burettes (except Class A glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Glass micro-syringes are considered the same as Class A glassware provided they are purchased with a manufacturer's certificate attesting to their accuracy. Micro-syringes are routinely purchased from Hamilton Company. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

Any device not regularly verified can not be used for any quantitative measurements.

## **20.4 Instrument Calibrations**

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration).

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed, if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

### **20.4.1 Calibration Standards**

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify

the number of calibration standards, a minimum of 3 calibration points (an exception includes ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or a vendor-certified different lot if a second source is not available). For unique situations, such as EPA 1653 analyses, where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

#### **20.4.1.1 Calibration Verification**

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used then bracketing standards are not required, only daily verifications are needed, unless specified by the reference method. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements. Refer to the specific SOPs for requirements. Most inorganic methods require the CCV to be analyzed after every 10 samples or injections, including matrix or batch QC samples.

**Note:** If an internal standard calibration is being used (e.g. GC/MS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument/method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

- a) when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or
- b) when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

#### **20.4.1.2 Verification of Linear and Non-Linear Calibrations**

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

#### **20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis**

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

#### **20.6 GC/MS Tuning**

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1. Instrumentation



TestAmerica Savannah Instrument List

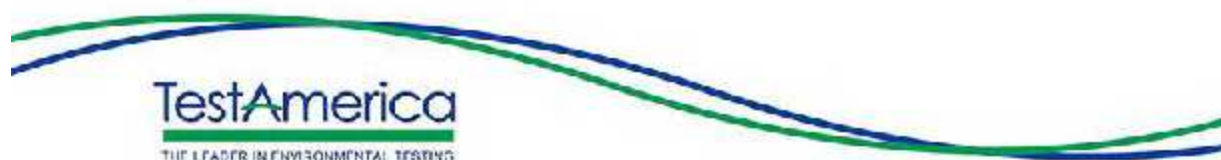
Equipment/ Instrument	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP	Varian (ICP E)	730-ES	IP0712M054	2008	New
ICP	Varian (ICP F)	730-ES	1P0803M118	2012	Used
ICP/MS	Agilent (ICP/MS A)	Agilent 7500C G3155A	JP 10300403	2002	New
ICP/MS	Agilent (ICP/MS B)	Agilent 7500CE G3272A	JP 14101289	2005	New
ICP/MS	Agilent (ICP/MS C)	Agilent 7700x G3281A	JP10390615	2011	New
CVAA	Leeman (2)	HYDRA AA II	00024	2011	New
GC/MS Semivolatiles	Hewlett- Packard (MS D)	5973/6890	US82311451	1999	New
GC/MS Semivolatiles	Hewlett- Packard (MS E)	5973/6890	US82311455	1999	New
GC/MS Semivolatiles	Hewlett- Packard (MS F)	5973/6890	US44647039	2004	New
GC/MS Semivolatiles	Hewlett- Packard (MS G)	5973/6890	US82311571	1999	New
GC/MS Semivolatiles	Hewlett- Packard (MS K)	5973/6890	CN10524062	2005	New
GC/MS Semivolatiles	Hewlett- Packard MS (N)	5973/6890	US72010580	1998	New
GC/MS Semivolatiles	Hewlett- Packard (MS R)	5973/6890N	21842170	2002	New
GC/MS Semivolatiles	Hewlett- Packard (MS T)	5973/6890	US33246115	2003	New
GC/MS Semivolatiles	Agilent (MS W)	5975/6890N	US10608004	2006	New
GC/MS Semivolatiles	Hewlett- Packard (MS X)	5975/6890N	CN10608061	2006	New
GC/MS Semivolatiles	Agilent (MS Y)	5975/7980A	US80838915	2008	New
GC/MS Volatiles	Hewlett- Packard (MS A)	5973/6890	US82311453	2000	New
GC/MS Volatiles	Hewlett- Packard (MS AA)	5973/6890N	US406220567	2013	Used

Rev. 05-2014

Page 1 of 3

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GC/MS Volatiles	Hewlett-Packard (MS B)	5973/6890	US82311452	2000	New
GC/MS Volatiles	Hewlett-Packard (MS C)	5975/7890	CN10917056	2012	New
GC/MS Volatiles	Hewlett-Packard (MS L)	5972/5890 II	3306A00159	1994	New
GC/MS Volatiles	Hewlett-Packard (MS M)	5972/5890 II	3251A00054	1992	New
GC/MS Volatiles	Hewlett-Packard (MS O)	5973/6890	US7200579	1993	New
GC/MS Volatiles	Hewlett-Packard (MS P)	5973/6890	US0039011	2000	New
GC/MS Volatiles	Hewlett-Packard (MS S)	5973/6890	US21843181	2002	New
GC/MS Volatiles	Agilent (MS U)	5973/6890	US52441057	2005	New
GC/MS Volatiles	Agilent (MSAB)	5975/6890	US80522680	2014	Transfer
GC/MS Volatiles	Agilent (MSAC)	5973/6890	US82311488	2014	Transfer
GC/MS Volatiles	Agilent (MSAD)	5973/6890	US82311485	2014	Transfer
Ion Chromatograph	Dionex (IC N)	ICS-1000	04060105		Used
Ion Chromatograph	Dionex (IC G)	ICS-2000	05101132	2005	New
Ion Chromatograph	Dionex (IC H)	ICS-2000	06080799	2006	New
Ion Chromatograph	Dionex (IC K)	ICS-2000	0307011	2012	Used
Ion Chromatograph	Dionex (IC L)	ICS-2000	05120486	2012	Used
GC Semivolatiles	Hewlett-Packard (SG J)	6890 (ECD)	US00033184	2000	New
GC Semivolatiles	Hewlett-Packard (SG K)	6890 (ECD)	US10223085	2002	New
GC Semivolatiles	Hewlett-Packard (SG L)	5890 II Plus (ECD)	3033A31398	2000	Used
GC Semivolatiles	Agilent (SG Q)	6890N (FID)	CN10521056	2005	New
GC Semivolatiles	Hewlett-Packard (SG S)	6890 Plus (ECD)	US00024188	2000	New
GC Semivolatiles	Hewlett-Packard (SG X)	6890N (ECD)	CN10406086	2003	New
GC Semivolatiles	Agilent (SG Y)	6890N (ECD)	CN10528081	2005	New



GC Semivolatiles	Agilent (SG Z)	6890N (ECD)	CN10814004	2008	New
GC Semivolatiles	Agilent (SGAA)	6890 (ECD)	US00031692	2013	Used
GC Semivolatiles	Agilent (SG AB)	6890N(FID)	US10224026	2013	Used
GC Volatiles	Agilent (VG G)	6890 (FID)	14921	2007	New
GC Volatiles	Agilent (VG U)	6890 (FID)	US10439011	2005	New
GC Volatiles	Agilent (VG V)	6890 (FID)	CN10619098	2006	New
GC Volatiles	Agilent (VG W)	6890 (FID)	CN10603131	2006	New
Liquid Chromatography	Hewlett-Packard (LC J)	1100	US72101013	2002	New
Liquid Chromatography	Hewlett-Packard (LC K)	1100	US72102590	2002	New
Liquid Chromatography	Hewlett-Packard (LC N)	1100	DE91607527	2008	Used
General Chemistry	Hach (TURB1)	2100 AN	950400000487	1995	New
General Chemistry	Lachat (1)	QuickChem 8000	A83000-1070	1997	New
General Chemistry	Lachat (2)	QC 8500 Series 2	100200001169	2010	New
General Chemistry	Lachat (3)	QuickChem 8000	A8300-1086	2012	Used
General Chemistry	Milton Roy Spectronic (SPC3)	301	20839	2009	Used
General Chemistry	Shimadzu	TOC-V CPN	H51404335036 CS	2006	New
General Chemistry	Mitsubishi	AOX-200	E7B20051	2011	New
General Chemistry	Mitsubishi	TOX-100	A7M42015	2005	New
General Chemistry	BOD AssayPlus	Version 3.0	270F6XB334	2006	New
General Chemistry	PCTitrate	Version 3.0	270G6XB370	2006	New
General Chemistry	Konelab (1)	Konelab20	M4218134	2000	New
General Chemistry	Konelab (2)	Konelab20	M3118114	2001	New

## **SECTION 21. MEASUREMENT TRACEABILITY**

### **21.1 Overview**

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, deionized (DI) water systems, automatic pipettes, and other volumetric measuring devices. (Refer to Section 20.3.) With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware and glass microliter syringes should be routinely inspected for chips, acid etching, or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

### **21.2 NIST-Traceable Weights and Thermometers**

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia-Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

A calibration laboratory's policy for achieving measurement traceability is defined and includes the subsequent elements of uncertainty. The calibration report or certificate contains a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. All calibration reports are filed in the QA Department.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

### **21.3 Reference Standards / Materials**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as EPA 1653 analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

#### **21.4      Documentation and Labeling of Standards, Reagents, and Reference Materials**

Reagents must be, at a minimum, the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. [Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained electronically. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily

available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within  $\pm 15\%$ , otherwise the certified values is used for the canister concentration.

**21.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's TALS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the TALS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- TALS Standard ID
- Special Health/Safety warnings if applicable



Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained electronically.

**21.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container
- Recommended Storage Conditions

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets, and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.



## **SECTION 22. SAMPLING**

### **22.1 Overview**

TestAmerica Savannah provides some sampling services. Sampling procedures are described in SOP SA-FD-05: *Field Sampling Procedures*.

### **22.2 Sampling Containers**

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness provided by the supplier are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

#### **22.2.1 Preservatives**

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

### **22.3 Definition of Holding Time**

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis.

### **22.4 Sampling Containers, Preservation Requirements, Holding Times**

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote, or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

### **22.5 Sample Aliquots / Subsampling**

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container,

the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*.

## **SECTION 23. HANDLING OF SAMPLES**

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

### **23.1 Chain of Custody (COC)**

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

#### **23.1.1 Field Documentation**

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time, and location of sampling
- Sample collector's name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to TestAmerica personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of

the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. When sampling personnel deliver the samples through a common carrier (e.g., Fed-Ex, UPS), the COC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date.

### **23.1.2     Legal / Evidentiary Chain-of-Custody**

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

## **23.2        Sample Receipt**

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

Additional information on the sample receipt process is given in SOP SA-CU-01: *Sample Receipt Procedures*.

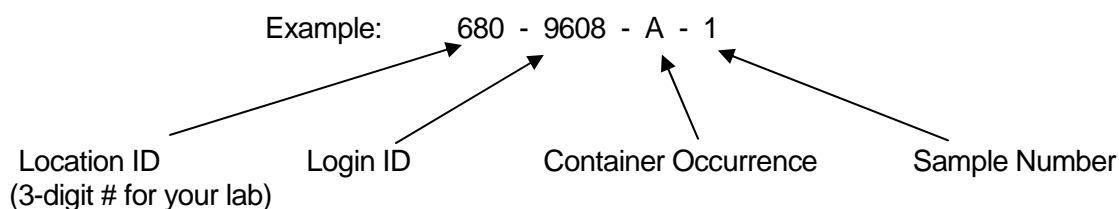
### **23.2.1     Laboratory Receipt**

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on the Sample Receipt Checklist in TALS and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

#### **23.2.1.1   Unique Sample Identification**

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at any time. This system includes identification for all samples, subsamples, and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Savannah is the laboratory (Location ID 680). The Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

Example: 680 - 9608 - A - 1 - A ← **Secondary Container Occurrence**

Example: 680-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1<sup>st</sup> occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

### 23.3 **Sample Acceptance Policy**

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the Project Manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

**23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and route them to the appropriate refrigerators or storage locations.

**23.3.2** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

#### **23.4      Sample Storage**

In order to avoid deterioration, contamination, or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers, or protected locations suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, storage blanks are maintained in the volatile sample refrigerators and analyzed every week.

Analysts retrieve the sample container allocated to their analysis from the designated storage location, prepare or analyze the sample, and return the remaining sample to the storage location from which it originally came. All samples are scanned into and out of the storage locations using the TALS sample custody program. Empty containers are scanned into the TALS sample custody program as empty and are properly disposed of. All samples are kept for at least 30 days after the report is sent out, which meets or exceeds most sample holding times. After this time, the samples are properly disposed of in accordance with the Environmental Health and Safety Manual.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

#### **23.5      Hazardous Samples and Foreign Soils**

Upon receipt, foreign soil samples are marked with a fluorescent green "FOREIGN SOIL" label prior to distributing to the analytical departments. Once the sample is received by the department, it is stored in a "FOREIGN SOIL ONLY" box segregated from other samples. Non-hazardous foreign soil samples are sent out for incineration by a USDA-approved waste



disposal facility. RCRA hazardous foreign soil samples are heat treated at the laboratory. After heat treatment, normal disposal procedures are followed. Refer to the Environmental Health and Safety Manual Addendum for additional information on disposal of hazardous samples. If not classified as hazardous, foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

### **23.6      Sample Shipping**

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they did not inadvertently omit a key part of regulatory compliance testing.

### **23.7      Sample Disposal**

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures outlined in the Savannah Addendum to the Environmental Health and Safety Manual. All procedures in the laboratory Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or

deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

## Chain of Custody (COC)

[illegible]

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## Figure 23-2. Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax, or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any TestAmerica facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).



### Sample Acceptance Policy

All samples will be evaluated against the criteria listed below. Samples which do not meet the criteria listed below will be qualified using the LIMS NCM Program and/or Sample Receipt Checklist.

- 1) Samples must arrive in good condition with a Chain-of-Custody filled out completely.
- 2) Samples must be properly labeled.
- 3) Samples must be in proper containers with adequate volume for the analysis.
- 4) Samples must be preserved according to the requirements of the requested analytical test method. Most analytical methods require chilling samples to 4°C. These criteria are met if the samples are chilled to below 8°C and above freezing. For methods with other temperature criteria (e.g. some bacteriological methods require  $\leq 8^{\circ}\text{C}$ ), the samples must arrive within  $\pm 2^{\circ}\text{C}$  of the required temperature or within the method specified range.

Note: Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- 5) Samples must be submitted with proper chemical preservation (pH) as required by the analytical test method.
- 6) Samples must be dechlorinated as required by the analytical test method.
- 7) Samples must be prepared and analyzed with the holding times defined in the analytical test method.
- 8) Samples submitted for Volatiles analyses must be submitted without headspace.

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**Figure 23-3. Login Sample Receipt Checklist**

**Login Sample Receipt Check List**

Client: TestAmerica Laboratories, Inc.

Job Number:

SDG Number:

Login Number:

List Source:

Creator:

List Number:

Question	T / F/ NA	Comment
Radioactivity either was not measured or, if measured, is at or below background		
The cooler's custody seal, if present, is intact.		
The cooler or samples do not appear to have been compromised or tampered with.		
Samples were received on ice.		
Cooler Temperature is acceptable.		
Cooler Temperature is recorded.		
COC is present.		
COC is filled out in ink and legible.		
COC is filled out with all pertinent information.		
There are no discrepancies between the sample IDs on the containers and the COC.		
Samples are received within Holding Time.		
Sample containers have legible labels.		
Containers are not broken or leaking.		
Sample collection date/times are provided.		
Appropriate sample containers are used.		
Sample bottles are completely filled.		
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs		
VOA sample vials do not have headspace or bubble is <6mm (1/4") in diameter.		
If necessary, staff have been informed of any short hold time or quick TAT needs		
Multiphasic samples are not present.		
Samples do not require splitting or compositing.		
Is the Field Sampler's name present on COC?		
Sample Preservation Verified		

TestAmerica Savannah

## SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

### 24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

### 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying, and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

### 24.3 Negative Controls

**Table 24-1. Example – Negative Controls**

Control Type	Details
Method Blank (MB)	<p>Used to assess preparation and analysis for possible contamination during the preparation and processing steps.</p> <p>The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.</p> <p>The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.</p> <p>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).</p> <p>Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.</p>
Calibration Blanks	Prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	Blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.



**Table 24-1. Example – Negative Controls**

Control Type	Details
Trip Blank <sup>1</sup>	Required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks <sup>1</sup>	Sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks <sup>1</sup>	Sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Holding Blanks	Referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

#### **24.4 Positive Controls**

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon method performance (e.g., Laboratory Control Sample), which entails both the preparation and measurement steps; and matrix effects (e.g., Matrix Spike or Sample Duplicate), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology, and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

##### **24.4.1 Method Performance Control - Laboratory Control Sample (LCS)**

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous

volatiles), or when all samples and standards undergo the same preparation and analysis process (such as phosphorus), a calibration verification standard is reported as the LCS.

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking as they cover the range of all of the Aroclors. Specific Aroclors may be used by request on a project specific basis.

## 24.5 Sample Matrix Controls

**Table 24-3. Sample Matrix Control**

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency <sup>1</sup>	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).

**Table 24-3. Sample Matrix Control**

Control Type	Details	
	Typical Frequency <sup>1</sup>	Added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSDs are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## 24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or surrogate spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary (recommended on an annual basis) unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm 3$  Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (CCV) unless the analytical method specifies a tighter limit.
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- For routine analytes that are not classified as poor performers, the lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable).
- If either the high or low end of the control limit changes by  $\leq 5\%$  from previous, the control chart may be visually inspected and, using professional judgment, they may be left unchanged if there is no effect on laboratory ability to meet the existing limits.

**24.6.1** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

The QA Department generates a Method Limit Group (MLG) in the TALS that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Savannah. The MLG includes an effective date and is updated each time new limits are generated and entered. Unless otherwise noted, limits within these tables are laboratory generated. The TALS maintains an archive of all limits used within the laboratory.

**24.6.2** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- If there are an allowable number of Marginal Exceedances (ME):

<11 analytes	0 marginal exceedances are allowed.
11 – 30 Analytes	1 marginal exceedance is allowed
31-50 Analytes	2 marginal exceedances are allowed
51-70 Analytes	3 marginal exceedances are allowed
71-90 Analytes	4 marginal exceedances are allowed
> 90 Analytes	5 marginal exceedances are allowed

Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).

Marginal exceedances should be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

**24.6.3** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.4** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client).

## **24.7 Additional Procedures to Assure Quality Control**

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 9 and 21.
- A discussion on selectivity of the test is included in Section 5.
- Constant and consistent test conditions are discussed in Section 18.
- The laboratories sample acceptance policy is included in Section 23.

## **SECTION 25. REPORTING RESULTS**

### **25.1 Overview**

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

### **25.2 Test Reports**

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is reviewed and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report) with a "Result" column header.

**25.2.2** Each report cover page includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. Job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

**25.2.5** The name and address of client and a project name/number, if applicable.

**25.2.6** Client project manager or other contact



**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

**25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**25.2.11** Reporting limits

**25.2.12** Method detection limits (if requested)

**25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets

**25.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.18** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Laboratory Director.

**25.2.19** When TNI accreditation is required, the laboratory shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

**25.2.20** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.21** When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

**25.2.22** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.23** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or preliminary report). A complete report must be sent once all of the work has been completed.

**25.2.24** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

**25.2.25** Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

### **25.3      Reporting Level or Report Type**

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including QC results.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

#### **25.3.1 Electronic Data Deliverables (EDDs)**

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Savannah offers a variety of EDD formats including Environmental Resources Program Information Management System (ERPIMS), Automated Data Review (ADR), Locus Focus (EIM), EQUIS ESBASIC, Environmental Quality Information Systems (EQUIS), Staged Electronic Data Deliverable (SEDD), EPA Region V EDD (EDMAN), and Terrabase.

EDD specifications are submitted to the IT department by the Project Manager for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

#### **25.4 Supplemental Information for Test**

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

#### **25.5 Environmental Testing Obtained From Subcontractors**

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP No. CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

#### **25.6 Client Confidentiality**

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore,

information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**25.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

*This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify sender immediately.*

## **25.7 Format of Reports**

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## **25.8 Amendments to Test Reports**

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained in the TALS, as is the original report. The revised report is stored in the project files under the sample number followed by "Rev#" where # is the number of the report revision.

When the report is re-issued, the revision number is placed on the cover/signature page of the report or at the top of the narrative page. A brief explanation of reason for the re-issue and a reference back to the last final report generated may be included.

## **25.9 Policies on Client Requests for Amendments**

### **25.9.1 Policy on Data Omissions or Reporting Limit Increases**

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

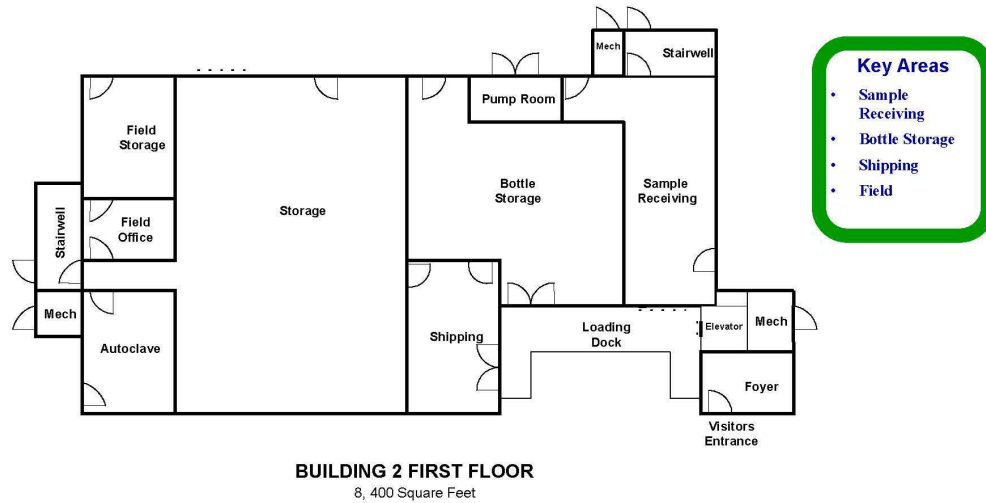
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

#### **25.9.2 Multiple Reports**

TestAmerica does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

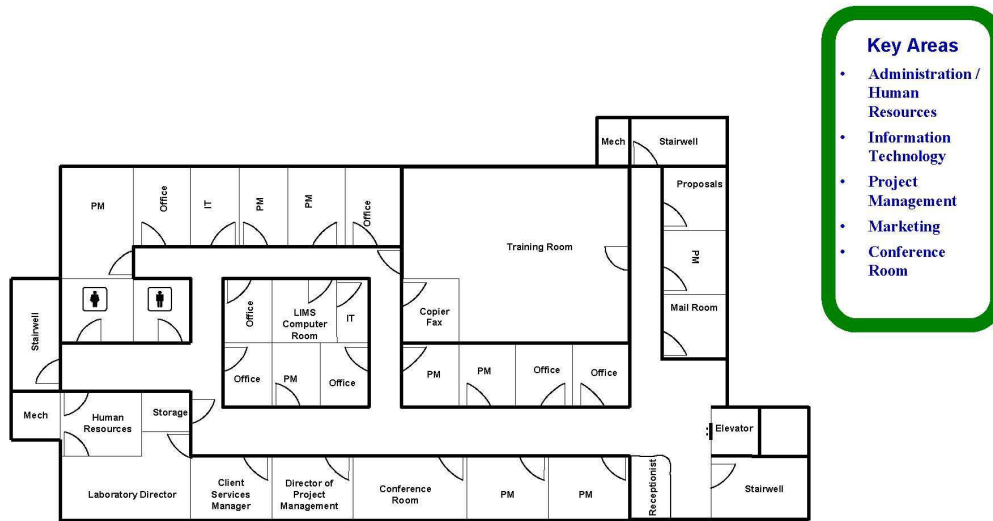
Appendix 1. Laboratory Floor Plan

## Floor Plan of Savannah





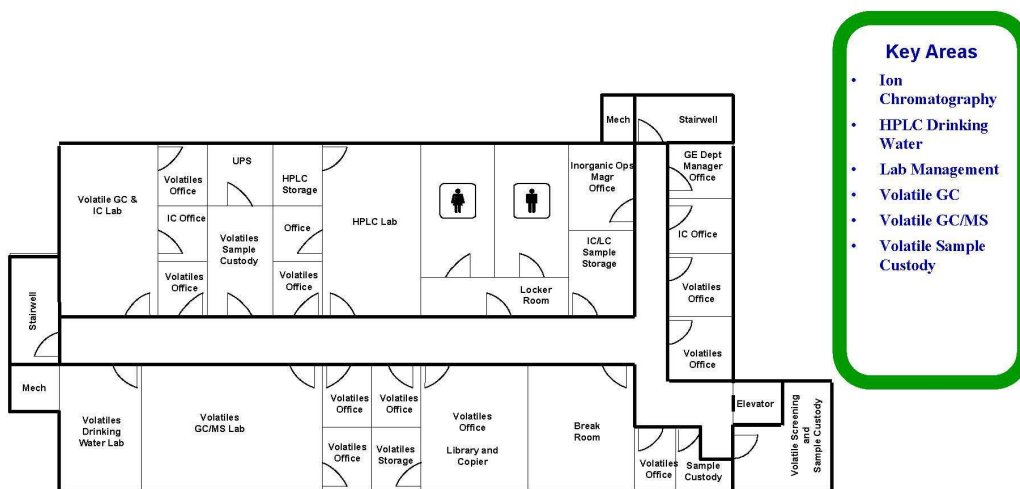
## Floor Plan of Savannah



**BUILDING 2 SECOND FLOOR**  
8,400 Square Feet

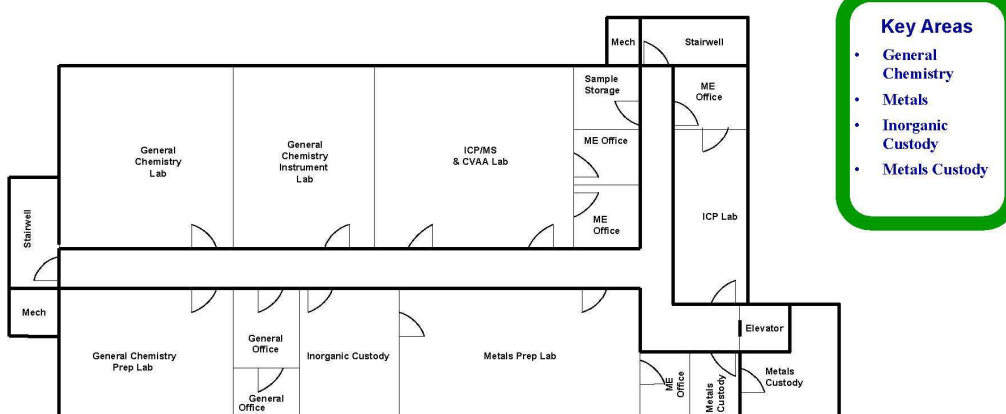
**TestAmerica**  
THE LEADER IN ENVIRONMENTAL TESTING

## Floor Plan of Savannah



**BUILDING 2 THIRD FLOOR**  
8,400 Square Feet

## Floor Plan of Savannah

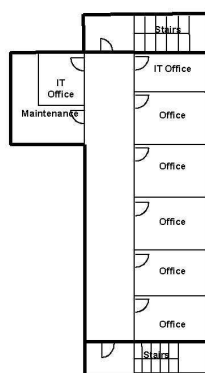


**BUILDING 2 FOURTH FLOOR**  
8,400 Square Feet

**TestAmerica**  
THE LEADER IN ENVIRONMENTAL TESTING



## Floor Plan of Savannah



**BUILDING 1 SECOND FLOOR**  
2,200 Square Feet

## **Appendix 2. Glossary/Acronyms (EL-V1M2 Sec. 3.1)**

### **Glossary:**

#### **Acceptance Criteria:**

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

#### **Accreditation:**

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (TNI)

#### **Accrediting Authority:**

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (TNI)

#### **Accuracy:**

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

#### **Aliquot:**

A representative portion of the sample, standard, or reagent.

#### **Analyst:**

The designated individual who performs the “hands-on” analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

#### **Analyte:**

The element, molecule, or compound that is being measured in a given procedure. Also referred to as a parameter.

#### **Analytical Method:**

Defines the sample preparation and instrumentation procedures that must be performed to determine the quantity of analyte in a sample.

#### **Analytical Sequence:**

The order in which calibration standards, verification standards, QC items, and samples are analyzed.

#### **Analytical Spike:**

Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

**Analytical Uncertainty:** A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)



**Anion:**

A negatively charged ion.

**Anomaly:**

Anomalous situations that are out of the ordinary but are not necessarily a method deviation and are not definitive enough to require a CAR are documented in the Non-Conformance Module. The use of the grand mean exception would require initiation of an Anomaly NCM.

**Aromatic:**

Relating to the six-carbon-ring configuration of benzene and its derivatives.

**Assessment:**

The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

**Assessment Team:**

The group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (TNI)

**Assessor:**

One who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (TNI)

**Audit:** A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

**Background Correction:**

A technique to compensate for variable background contribution to the instrument signal and the determination of trace metals.

**Batch:**

Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

**Bias:**

The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

**Blank:**

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

**Blind Sample:**

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

**Calibration:**

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (TNI)

**Calibration Check Compounds (CCC):**

Term used in conjunction with SW-846, Method 8260 and 8270 to refer to the compounds in which the percent RSD is evaluated against method-prescribed criteria to decide the validity of a calibration.

**Calibration Curve:**

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

**Calibration Method:**

A defined technical procedure for performing a calibration. (TNI)

**Calibration Standard:**

A substance or reference material used to calibrate an instrument.

**Certified Reference Material (CRM):**

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30-2.2)

**Chain of Custody:**

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (TNI)

**Cation:**

A positively charged ion.

**Chemical Analysis:**

Any of a variety of laboratory methods used to evaluate the concentrations of compounds and elements present in an environmental sample.

**Clean Air Act:**

The enabling legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them.

**Client Complaint:**

A complaint is a situation where dissatisfaction is expressed with the service provided by the laboratory.

**Composite Sample:**

Portions of material collected from more than one spatial location or at different times that are blended and submitted for chemical analyses. Composite samples can provide data representative of a large area with relatively few samples. However, the resulting data are less accurate with regard to the concentrations of contaminants detected in a specific location, because they represent average values.

**Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):**

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

**Compromised Samples:**

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (TNI)

**Concentration:**

The mass of analyte per unit mass or volume of sample. Common units of concentration for environmental analyses are microgram per liter or kilogram (ug/L or ug/kg) and milligrams per liter or kilogram (mg/L or mg/kg).

**Confidence interval:**

For normally distributed (random) data, the intervals where 68%, 95%, and 99% of the data fall. 68% of the data should fall within 1 standard deviation of the mean, 95% of the data should fall within 2 standard deviations of the mean, and 99% of the data should fall within 3 standard deviations of the mean.

**Confidential Business Information (CBI):**

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

**Confirmation:**

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional Cleanup procedures

(TNI)

**Conformance:**

An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

**Continuing Calibration Verification (CCV) Standard:**

A mid-concentration analytical standard run periodically to verify the calibration of the analytical instrument. Also known as continuing calibration check (CCC).

**Contract Laboratory Program (CLP):**

A nationwide laboratory network established by the USEPA, structured to provide legally defensible analytical results to support USEPA enforcement actions or other requirements of the use community. The CLP incorporates a level of quality assurance appropriately designed for the intended usage of the data.

**Control Limits:**

Accuracy or precision ranges that determine whether the experimentally determined results are in control. If the results are within the acceptance ranges, the results are said to be in control; if the results are outside the limits, they are said to be out-of-control.

**Corrective Action:**

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

**Corrective Action Report (CAR):**

The CAR form is used in situations where a recurring problem or breakdown in systems is observed and warrants a more thorough investigation than a single-event NCR. CARs may be initiated from: a specific nonconformance situation (NCM), an observed trend or frequency of events that warrant corrective action, an audit finding, etc.

**Correlation Coefficient:**

A number ( $r$ ), which indicates the degree of dependence between two variables (concentration and response). The more dependent the variables are, the closer the value is to one. This value is used to evaluate the straightness of a line, (the linearity of the instrument).

**Data Audit:**

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (TNI)

**Data Reduction:**

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

**Data Validation:**

An evaluation of laboratory data quality based on a review of the data deliverables. This process involves procedures verifying instrument calibration, calibration verification, and other method-specific performance criterion.

**Deficiency:**

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

**Demonstration of Capability (DOC):**

Procedure to establish the ability to generate acceptable accuracy and precision. This is done initially upon starting a new method and then continues each year the method is performed.

**Detection Limit:**

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (TNI)

**Direct Aqueous Injection (DAI):**

A technique in which an aliquot of the aqueous sample or aqueous leachate is injected directly into the gas chromatograph with no prior sample preparation.

**Disposal:**

Final placement or destruction of wastes. Disposal may be accomplished through the use of landfills, treatment processes, etc.

**Document Control:**

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

**Duplicate Analyses:**

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

**E. coli:**

Bacteria giving a positive total coliform response and possessing the enzyme B-glucuronidase, which cleaves the fluorogenic substrate MUG, resulting in the release of a fluorescent product when viewed under long-wavelength UV light.

**Environmental Detection Limit (EDL):**

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (TNI Radioanalysis Subcommittee)

**Equipment Blank:**

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)

**External Standard Calibration:**

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

**Extractable Organics:**

Semivolatiles (base/neutral and acid extractable compounds) and pesticide/polychlorinated biphenyl compounds that can be partitioned into an organic solvent from the sample matrix and are amenable to gas chromatography (GC).

**Fecal Coliforms:**

A subset of total coliforms that grow and ferment lactose at an elevated incubation temperature (44.5°C) and are also referred to as thermotolerant coliforms. Fecal coliforms produce colonies that appear in various shades of blue, domes and glistening, ranging in size from pinpoints to several millimeters. This group consists of mostly E. Coli (EC) but also includes some other enterics. Fecal coliforms are a more specific indicator organism for contamination. This type of bacteria is associated with the fecal material of warm-blooded animals.

**Federal Insecticide, Fungicide and Rodenticide Act (FIFRA):** The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (TNI)

**Federal Water Pollution Control Act (Clean Water Act, CWA):**

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

**Field Blank:**

Blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)



**Field Control Samples:**

General term assigned to field-generated replicates (duplicates/splits/spikes), blanks, background/upgradient samples, etc.

**Field Duplicate Sample:**

Independent sample collected at approximately the same time and place, using the same methods as another sample. The duplicate and original sample are containerized, handled, and analyzed in an identical manner.

**Field of Testing:**

TNI's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (TNI)

**Filtrate:**

A filtered liquid.

**Filtration:**

The physical removal of solid particles from a liquid wastestream by passing the liquid across a filter medium, which serves as a barrier to the solid material.

**Finding:**

An assessment conclusion that identifies a condition having a significant effect on an item or activity. As assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (TNI)

**Gas Chromatography/Mass Spectroscopy (GC/MS):**

Two distinct analytical techniques used to separate and identify organic compounds: the GC is used for the separating portion and the MS is used as the detection portion of an analysis. Both techniques are typically performed by a single instrument.

**Good Laboratory Practices (GLP):**

Formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729.

**Heavy Metals:**

In reference to environmental sampling, typically identified as the following trace inorganics: cadmium, lead, mercury, silver, etc. (all metals of health concern). Heavy metals can cause biological damage if consumed at low concentrations and tend to accumulate in the food chain.

**Heterotrophic Bacteria:**

A large group of bacteria that obtain energy by oxidizing organic matter. Coliform bacteria are a subset of this group.

**Holding Times (Maximum Allowable Holding Times):**

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

**Homogeneous:**

The quality of uniform composition.

**Initial Calibration Verification (ICV):**

A mid-concentration analytical standard run immediately after the calibration to verify the calibration of the analytical instrument. Also known as initial calibration check (ICC).

**Inorganic Chemicals:**

Chemical substances of mineral origin, not of basically carbon structure.

**Inquiry:**

A question or request for information about the service provided by the laboratory.

**Inspection:**

An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

**Instrument Blank:**

A blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

**Instrument Detection Limit (IDL):**

The minimum amount of a substance that can be measured on a specific instrument, with a specified degree of confidence that the amount is greater than zero. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. An IDL value, by definition, has an uncertainty of  $\pm 100\%$ . The IDL thus represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

**Instrument Performance Check Solution (IPC):**

A solution of one or more method analytes, surrogates, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

**Intermediate or Secondary Stock Standard:**

A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.

**Internal Standard:**

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

**Internal Standard Calibration:**

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

**Instrument Blank:**

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

**Instrument Response:**

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

**Job number:**

A sequential number that is assigned to each client's samples upon receipt into the laboratory. This log number provides the primary means of associating the samples to the client.

**Laboratory:**

A defined facility performing environmental analyses in a controlled and scientific manner. (TNI)

**Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):**

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (TNI)

**Laboratory Duplicate:**

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (TNI)

**Laboratory Fortified Blank (LFB):**

An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required method detection limit. The percent recovery (accuracy) result for the LFB must fall within the limits listed in the TALS. Also referred to as a laboratory control standard (LCS).

**Laboratory Fortified Sample Matrix (LFM):**

An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The percent recovery (accuracy) result for the LFM must fall within the limits listed in the TALS. Also referred to as a matrix spike (MS).

**Laboratory Fortified Sample Matrix Duplicate (LFMD):**

A replicate laboratory fortified sample matrix.

**Laboratory Performance Check Solution (LPC):**

A solution of selected method analytes used to evaluate the performance of the instrumental system with respect to a defined set of method criteria.

**Laboratory Quality Manual (LQM):**

A document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system. Also referred to as the Quality Assurance Manual (QAM) or Quality Assurance Plan (QAP).

**Laboratory Reagent Blank (LRB):**

An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Also referred to as a method blank (MB).

**Leachate:**

The liquid portion of a sample that passes through a 0.6 $\mu$ m filter in the initial evaluation of the percent solids, or the liquid that passes through a 0.6 $\mu$ m filter after the sample has been subjected to the TCLP. The liquid produced by subjecting the sample to the SPLP method.

**Least Squares Regression (1<sup>st</sup> Order Curve):**

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

**Limit of Detection (LOD):**

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

**Liquid phase:**

The portion of the sample that passes through the 0.6-0.8µm filter when subjected to a pressure of 50psi during the TCLP or SPLP process.

**Manager (however named):**

The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (TNI)

**Mass Spectrometry (MS):**

A detection instrument that differentiates compounds by their differences in mass, or mass fragments. The basic components of the MS are the ion source and lenses, the mass filter (quadrupoles), and the electron multiplier. The ion source and lenses create the ions and propel them on a consistent path to the quadrupoles. The quadrupoles filter the ions that are produced in the source, allowing them to continue to the electron multiplier, where the ions are collected and the signal sent to the data system.

**Mass Spectra:**

A graphical representation of the abundance of the mass ions produced when a compound is detected by mass spectrometry. The mass spectrum is essentially a fingerprint of the compound and along with the retention time of the compound provides excellent qualitative information about the presence of the compound.

**Matrix:**

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

**Aqueous:** Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

**Drinking Water:** any aqueous sample that has been designated as a potable or potential potable water source.

**Saline/Estuarine:** any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

**Non-aqueous Liquid:** any organic liquid with <15% settleable solids.

**Biological Tissue:** any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

**Solids:** includes soils, sediments, sludges, and other matrices with >15% settleable solids.

**Chemical Waste:** a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

**Matrix Duplicate (MD):**

Duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

**Matrix Spike (spiked sample or fortified sample):**

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

**Matrix Spike Duplicate (spiked sample or fortified sample duplicate):**

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

**Method Blank:**

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (TNI)

**Method Detection Limit:**

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

The MDL is defined as:

$$MDL = SD \otimes t(0.99)$$

SD = standard deviation of the replicates

t(0.99) = Student's t-Value at the 99% confidence level for number of replicates



**Most Probable Number (MPN):**

An estimate of the mean density of coliforms in a sample based on certain probability formulas.

**National Environmental Laboratory Accreditation Conference (NELAC):**

A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (TNI)

**National Environmental Laboratory Accreditation Program (NELAP):**

The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (TNI)

**Neat standard:**

A pure compound, element, or salt that contains the target analyte. The purity, usually expressed as a percent, of the neat standard must be known. Also known as a source reagent in TALS.

**Negative Control:**

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (TNI)

**NELAC Standards:**

The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (TNI)

**Non-conformance:**

Any occurrence that prevents the lab from delivering data that is compliant with the control criteria published (or incorporated by reference) in an applicable QA plan. The Non-conformance Module is used to document nonconformance conditions and to specify the necessary action(s) taken to correct the specific problem.

**Organic:**

Referring to or derived from living organisms; any compound containing carbon.

**Parts Per Billion (ppb):**

One part of analyte per billion parts of sample. For aqueous samples, a ppb is equivalent to ug/L; for soils, ug/kg.

**Parts Per Million (ppm):**

One part of analyte per million parts of sample. For aqueous samples, a ppm is equivalent to mg/L; for soils, mg/kg.

**Peak Gaussian Factor (PGF):**

A means to measure peak symmetry and monitoring retention time drift over time. Critically evaluate peak in the instrument performance check sample, and calculate the PGF as follows,

$$PGF = \frac{1.83 \otimes W(1/2)}{W(1/10)}$$

where:

W(1/2) is the peak width at half height

W(1/10) is the peak width at tenth height

Percent Recovery:

Percent recovery is used to assess accuracy and is calculated:

$$\%REC = \frac{C_{\text{experimental}}}{C_{\text{known}}} \otimes 100$$

where:

C<sub>experimental</sub> = experimentally determined concentration

C<sub>known</sub> = known or theoretical concentration

Percent Solids:

The proportion of solid in a soil sample determined by drying an aliquot of the sample.

Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (TNI)

Performance Based Measurement System (PBMS):

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (TNI)

pH:

A numerical designation of relative acidity or basicity (Alkalinity). A pH of 7 indicates neutrality; lower values indicate increasing acidity; high values indicate increasing alkalinity.

Precision:

The agreement between two or more experimentally determined results. Precision is routinely expressed as the relative percent difference between two results. Precision is not routinely used as a measurement to determine if the analysis is in control but may be required for certain programs and agencies.

Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

Post-Digestion Spike:

Addition of a known concentration of analyte to an aliquot of sample after the preparation steps have been performed.

**Preservation:**

Refrigeration and or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample. Methods used to retard degradation of chemical analytes within samples by inhibiting decomposition by biological action, chemical reactions, and reducing sorption effects. Methods include limiting headspace, chemical, acid, or base addition, protection from light, cooling, etc.

**Precision:**

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

**Preservation:**

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (TNI)

**Preventive Action:**

The pro-active process of noting and correcting a potential problem before it happens due to a weakness in a system, method, or procedure.

**Procedural Standard Calibration:**

A calibration method where aqueous calibration standards are prepared and processed (e.g., purged, extracted, and/or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiency in the processing procedure.

**Proficiency Testing:**

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

**Proficiency Testing Program:**

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

**Proficiency Test Sample (PT):**

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

**Quality Assurance:**

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

**Quality Assurance [Project] Plan (QAPP):**

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

**Quality Control:**

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

**Quality Control Sample:**

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

**Quality Manual:**

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

**Quality System:**

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

**Quantitation Limit (QL):**

The lowest point at which a substance can be quantitatively measured with a specified degree of confidence using a specific method. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL) or Reporting Limit (RL).

**Quantitation Limits:**

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (TNI)

**Range:**

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

**Raw Data:**

Any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports

specifying inclusion of “raw data” do not need all of the above included, but sufficient information to create the reported data.

**Reagent:**

A material that is used in a process or analysis but is not directly related to the measured analyte concentration.

**Reagent Blank (method reagent blank):**

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

**Reference Material:**

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

**Reference Method:**

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (TNI)

**Reference Standard:**

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

**Regulatory Threshold Limit:**

The concentration of analyte in the TCLP leachate at which the sample is deemed hazardous.

**Relative Percent Difference:**

The relative percent difference is calculated between the concentrations of two spikes or sample duplicates:

$$\%RPD = \left| \frac{(C_1 - C_2)}{\frac{C_1 + C_2}{2}} \right| \otimes 100$$

Where:

$C_1$  = concentration of the sample or spike

$C_2$  = concentration of the sample duplicate or spike duplicate

**Replicate Analyses:**

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (TNI)

**Reporting Limit (RL):**

Defines the lowest concentration that can be reported with reasonable certainty that the result falls within the laboratories' accuracy and precision limits. Also referred to as the practical quantitation limit or PQL, the RL is usually defined as the lowest point in the calibration curve or the sample equivalent concentration of the lowest point in the calibration curve.

**Representativeness:**

A qualitative measure of the extent to which a sample(s) acquired from a medium describes the chemical characteristics of that medium.

**Requirement:**

Denotes a mandatory specification; often designated by the term "shall". (TNI)

**Resolution:**

Also known as separation, or percent resolution. The separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smallest peak being resolved, and multiplied by 100.

**Resource Conservation and Recovery Act (RCRA):**

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (TNI)

**Safe Drinking Water Act (SDWA):**

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (TNI)

**Sample:**

A portion of material collected for chemical analyses. Note that a sample is identified by a unique sample number and that the term and the number may apply to multiple sample containers, if a single sample is submitted for a variety of chemical analyses.

**Sample Duplicate:**

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

**Sampling and Analysis Plan (SAP):**

A formal document describing the detailed sampling and analysis procedures for a specific project.

**Second Order Polynomial Curve (Quadratic):** The 2<sup>nd</sup> order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.990.

**Secondary or Intermediate Stock Standard:**

A solution made from two or more stock standards. A secondary standard may also be a certified solution purchased from a vendor as a mixture of several target analytes. Also known as a source reagent in TALS if purchased and an intermediate reagent if prepared in the lab.



**Selectivity:**

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

**Semivolatile Organics:**

Compounds that are amenable to analysis by extraction of the sample with an organic solvent. The term semivolatile organic is used synonymously with base/neutral/acid (BNA) compounds.

**Sensitivity:**

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

**Solvent:**

The organic liquid used to extract the compounds of interest out of the sample matrix. The solvent is also used to dissolve (put into solution) standards. In general, the solvent used to prepare the standards is also used to extract the samples. A good rule of thumb is that "like dissolves like", that is, a solvent must be similar in chemical structure to the compound that is being extracted or being dissolved. For most organic extractions, the solvent should also not be miscible (dissolves in all proportions) with water.

**Spike:**

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period. (TNI)

**Standard:**

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of TNI and meets the approval requirements of NELAC procedures and policies. (ASQC)

**Standard Operating Procedures (SOPs):**

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

**Standardized Reference Material (SRM):**

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

**Stock standard:**

A solution made from one or more neat standards. The stock standard will usually have a high concentration, usually higher than 1000mg/L (1000ug/mL). This standard can also be purchased from a certified vendor. Also known as a source reagent in TALS.

**Storage Blank:**

A blank matrix stored with field samples of a similar matrix.

**Supervisor (however named):**

The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (TNI)

**Surrogate:**

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

**Suspended Metals:**

The concentration of metals determined in the portion of a sample that is retained on a 0.45- $\mu$ m filter. (The concentration of suspended metals may also be calculated from the difference between the total metals sample results minus the dissolved metals sample results.)

**Systems Audit (also Technical Systems Audit):**

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

**System Performance Check Compounds (SPCCs):**

Term used in conjunction with SW-846, Method 8260 and 8270, to refer to the compounds in which the response factor (RF) is evaluated against method-prescribed criteria to decide the validity of a calibration.

**Target Analyte List (TAL):**

Refers to the Contract Lab Program (CLP) list of inorganic analytes that includes metals and cyanide. May also refer to any general list of inorganic target analytes.

**Target Compound List (TCL):**

Refers to the Contract Lab Program (CLP) list of organic compounds that includes volatiles (GC/MS), semivolatiles (GC/MS), and pesticides and PCBs (GC/EC). May also refer to any general list of organic target compounds.

**Technical Director:**

Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (TNI)

**Test:**

A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

**Test Method:**

An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (TNI)

**Total Coliforms:**

Gram-negative, facultative anaerobic rod-shaped enteric bacteria that ferment lactose to produce colonies with a metallic sheen (yellow to green) when viewed under a fluorescent lamp or acid and gas within 48 hours incubated at 35°C. All bacteria possessing the enzyme B-D-galactosidase, which cleaves the chromogenic substrate ONPG, resulting in release of a chromogen that produces a color change in the sample. They are used as an indicator of contamination in samples although some total coliform bacteria are found naturally in environmental samples. This type of bacteria is commonly found in the intestines of humans.

**Total Metals:**

Concentration of metals determined in an unfiltered water sample which is preserved (acidified) in the field, transported to the laboratory, and then follows a rigorous digestion.

**Total Recoverable Metals:**

Concentration of metals in an unfiltered water sample which is preserved (acidified) in the field and transported to the lab, which then performs the digestion with hot dilute mineral acid. This preparation method is typically utilized for drinking water samples and TCLP extracts.

**Toxic Substances Control Act (TSCA):**

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (TNI)

**Traceability:**

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

**Trip Blank:**

Samples prepared by adding clean, analyte-free water to sample containers for analysis for volatile organics. Preservatives are added to the blank, and the containers are sealed prior to the sampling trip. Trip blanks are transported with empty sample containers to the site of work and remain sealed until analyzed with collected environmental samples. Trip blanks permit evaluation of contamination generated from sample containers or occurring during the shipping and laboratory storage process.

**Tune:**

To adjust the parameters of the mass spectrometer in order to meet the mass calibration criteria.

**Uncertainty:**

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

**United States Environmental Protection Agency (EPA):**

The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

**Validation:**

The process of substantiating specified performance criteria. (EPA-QAD)

**Verification:**

Confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

**NOTE:** In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

**Volatile Organic Compound (VOC):**

An organic compound that is amenable to purge and trap analysis. In general, VOC have low boiling points (<200°C), high vapor pressures (tend to evaporate easily at low temperatures), and have low molecular weight (generally less than 300amu).

**Work Cell:**

A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

**Working Standard:**

The standard that is analyzed on the instrument or using the analytical procedure. Also known as an intermediate reagent in TALS.

**Acronyms:**

ACRONYM	DEFINTION
A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
AFCEE	Air Force Center for Environmental Excellence
AL	Action Level
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
bgs	Below Ground Surface
BNA	Base, Neutral, Acids (Semivolatile Organics)
BOD	Biochemical Oxygen Demand
BS	Blank Spike
BSD	Blank Spike Duplicate
BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
BTU	British Thermal Unit
CA	Corrective Action
CAA	Clean Air Act
CAR	Corrective Action Report
CBOD	Carbonaceous Biochemical Oxygen Demand
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CDC	Continuing Demonstration of Capability
CDOC	Continuing Demonstration of Capability
CDQO	Chemical Data Quality Objective
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act

ACRONYM	DEFINTION
MRF	Method Request Form
MRL	Method Reporting Limit
MS	Mass Spectrometer
MS	Matrix Spike
MS/MS	Tandem Mass Spectrometry
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
MW	Monitoring Well
NBS	National Bureau of Standards
NCASI	National Counsel for Air and Stream Improvement, Inc.
NCM	Non-Conformance Module
NCR	Non-Conformance Report
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
nm	Nanometer
NPD	Nitrogen – Phosphorus Detector
NPDES	National Pollutant Discharge Elimination System
NPW	Non-Potable Water
ORO	Oil Range Organics
OSHA	Occupational Safety and Health Administration
OSTR	Outstanding SOP Training Report



ACRONYM	DEFINTION
CF	Calibration Factor
CFR	Code of Federal Regulations
CLLE	Continuous Liquid-Liquid Extraction
CLP	Contract Laboratory Program
COA	Certificate of Analysis
COC	Chain of Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRF	Change Request Form
CRQL	Contract Required Quantitation Limit
CSM	Corporate Safety Manual
CU	Custody
CVAA	Cold Vapor Atomic Absorption
CWA	Clean Water Act
DAI	Direct Aqueous Injection
DFTPP	Decafluorotriphenylphosphate
DM	Department Manager
DO	Dissolved Oxygen
DOC	Demonstration of Capability
DOD	Department of Defense
DOD QSM	Department of Defense Quality Systems Manual
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
DRO	Diesel Range Organics
DU	Duplicate
DUP	Duplicate
DW	Drinking Water
ECD	Electron Capture Detector
EDD	Electronic Data Deliverable
EDQM	Environmental Data Quality Management
EHS	Environmental Health and Safety
EHSM	Environmental Health and Safety Manual

ACRONYM	DEFINTION
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PCB	Polychlorinated Biphenyl
PDA	Photodiode Array
PDS	Post Digestion Spike
PE	Performance Evaluation
PGF	Peak Gaussian Factor
PID	Photoionization Detector
PM	Project Manager
PNA	Polynuclear Aromatic Hydrocarbon
PP	Project Plan
ppb	Parts Per Billion
PPE	Personnel Protective Equipment
PPL	Priority Pollutant List
ppm	Parts Per Million
ppq	Part Per Quadrillion
ppt	Parts Per Trillion
PQL	Practical Quantitation Limit
PRG	Preliminary Remediation Goals
PT	Proficiency Test
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl Chloride
PW	Potable Water
PWS	Public Water System
QA	Quality Assurance
QAM	Quality Assurance Manager
QAM	Quality Assurance Manual
QAMP	Quality Assurance Management Plan
QAN	Quality Assurance Navigator
QAP	Quality Assurance Plan
QAPjP	Quality Assurance Project Specific Plan
QAPP	Quality Assurance Project Plan
QAS	Quality Assurance Specialist

ACRONYM	DEFINTION
ELCD	Electrolytic Conductivity Detector
EPA	U.S. Environmental Protection Agency
ERPIMS	Environmental Resources Program Information Management System
eV	Electron Volt
FID	Flame Ionization Detector
FPD	Flame Photometric Detector
GALP	Good Automated Laboratory Practices
GC	Gas Chromatograph or Gas Chromatography
GC/MS	Gas Chromatograph/Mass Spectrometer
GE	General
GFAA	Graphite Furnace Atomic Absorption
GLP	Good Laboratory Practices
GPC	Gel Permeation Column (Gel Permeation Chromatography)
GRO	Gasoline Range Organics
HAA	Haloacetic Acids
HAPS	Hazardous Air Pollutants
HAZMAT	Hazardous Materials
HDPE	High Density Polyethylene
HECD	Electrolytic Conductivity Detector
HPLC	High Performance Liquid Chromatography
HRGC/HRMS	High Resolution Gas Chromatography/Hugh Resolution Mass Spectrometry
HT	Holding Time
HTRW	Hazardous, Toxic, and Radioactive Waste
HTV	Holding Time Violation
IC	Ion Chromatography
IC/EC	Ion Chromatography/Electric Conductivity
IC/MS	Ion Chromatography/Mass Spectrometer

ACRONYM	DEFINTION
QC	Quality Control
QCS	Quality Control Sample
QCSR	Quality Assurance Summary Report
QL	Quantitation Limit
QMP	Quality Management Plan
QSM	Quality Systems Manual
RCRA	Resource Conservation Recovery Act
RF	Response Factor
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RT	Retention Time
RTW	Retention Time Window
SAP	Sampling and Analysis Plan
SARA	Superfund Amendments and Reauthorization Act
SD	Standard Deviation
SD	Sample Dilution
SD	Sample Duplicate
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SG	Semi Volatile Gas Chromatography
SIM	Selected Ion Monitoring
SM	Semi Volatile Mass Chromatography
SOC	Synthetic Organic Compound

ACRONYM	DEFINTION
ICAP	Inductively Coupled Argon Plasma Emission Spectroscopy
ICB	Initial Calibration Blank
ICCS	Interference Calibration Check Sample
ICOC	Internal Chain of Custody
ICP	Inductively Coupled Plasma
ICP/MS	ICP/Mass Spectrometer
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDC	Initial Demonstration of Capability
IDL	Instrument Detection Limit
IDOC	Initial Demonstration of Capability
IH	Industrial Hygiene
IPC	Instrument Performance Check Standard
IR	Infrared Radiation
IS	Internal Standard
ISO	International Standards Organization
ISTD	Internal Standard
LC	Liquid Chromatography
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LFB	Laboratory Fortified Blank
LFM	Laboratory Fortified Matrix
LFMD	Laboratory Fortified Matrix Duplicate
LIMS	Laboratory Information Management System
LM	Laboratory Manager
LOD	Limit of Detection
LOQ	Limit of Quantitation
LPC	Laboratory Performance Check
LQM	Laboratory Quality Manual
LRB	Laboratory Reagent Blank
LUFT	Leaking Underground Fuel Tank
LUST	Leaking Underground Storage Tank

ACRONYM	DEFINTION
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compound
SPE	Solid Phase Extraction
SPLP	Synthetic Precipitation Leaching Procedure
SR	Shipping and Receiving
SRM	Standard Reference Material
SS	Suspended Solids
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOC	Semi Volatile Organic Compound
SW-846	Solid Waste Analytical Protocols
TAL	Target Analyte List
TALS	TestAmerica LIMS System
TAT	Turn-Around-Time
TCL	Target Compound List
TCLP	Toxicity Characteristic Leachate Procedure
TDS	Total Dissolved Solids
TEPH	Total Extractable Petroleum Hydrocarbons
THM	Trihalomethanes
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TM	Technical Manager
TOC	Total Organic Carbon
TOX	Total Organic Halides
TPH	Total Petroleum Hydrocarbons
TRPH	Total Recoverable Petroleum Hydrocarbons
TS	Total Solids
TSD	Thermionic Specific Detector
TSS	Total Suspended Solids
TVPH	Total Volatile Petroleum Hydrocarbons
TVS	Total Volatile Solids
UCL	Upper Confidence Level

ACRONYM	DEFINTION
MB	Method Blank
MB	Microbiology
MBAS	Methylene Blue Active Substances
MCL	Maximum Contaminant Level
MCT	Maximum Conductivity Threshold
MD	Matrix Duplicate
MDL	Method Detection Limit
ME	Metals
µg/L	Microgram per Liter
mg/L	Milligram per Liter
MLG	Method Limit Group
µm	Micrometer
MPN	Most Probable Number

ACRONYM	DEFINTION
UCMR	Unregulated Contaminant Monitoring Rule
US EPA	United States Environmental Protection Agency
USACE	United States Army Corps of Engineers
USDA	United States Department of Agriculture
USGS	United States Geological Service
UST	Underground Storage Tank
UV	Ultraviolet
VG	Volatile Gas Chromatography
VM	Volatile Mass Chromatography
VOA	Volatile Organic Analysis / Volatile Organic Analyte
VOC	Volatile Organic Compound
ZHE	Zero Headspace Extraction

### **Appendix 3. Laboratory Certifications, Accreditations, Validations**

TestAmerica Savannah performs work from clients located throughout the United States, as well as in some foreign countries. Most states and/or federal agencies maintain a laboratory accreditation program that requires a laboratory to obtain certification with their agency. To obtain certification, a laboratory must maintain an effective quality system that meets the requirements of the agency. Common components of the quality system requirements include maintaining up-to-date standard operating procedures (SOPs) and a Quality Assurance Manual (QAM); participating in a Proficiency Testing (PT) program; performing method detection limit (MDL) studies, initial and continuing demonstrations of capability studies (IDOCs/CDOCs), and internal assessments; and completing an annual renewal application. In addition to the requirements needed for certification, many agencies have specific analytical and/or reporting requirements that laboratories must follow.

Many agencies offer certification via reciprocity. Reciprocity is the acknowledgement of another state and/or agency's certification program. The most common types of reciprocity are homestate reciprocity and TNI (NELAC) reciprocity.

Lab Management, Project Management, Sales & Marketing, and the QA Manager may initiate requests for certification or accreditation. The QA staff completes the administrative tasks associated with the application and maintains the related documents in accordance with SOP SA-QA-001: *Document Control Program*.

Laboratory management has the responsibility and authority to ensure that laboratory operations are in compliance with program and regulatory requirements of the jurisdiction for which laboratory certification/accreditation is sought and maintained.

To perform compliance work in a particular state, the laboratory must maintain certification for the reported analytes. Most accrediting authorities will certify laboratories on a matrix/method/analyte level. For example:

Soil / EPA 8260B / benzene  
Water / EPA 624 / toluene

Generally, laboratories must apply and submit supporting documentation (SOPs, MDLs, IDOCs, PTs, etc.) for each individual matrix/method/analyte combination.

#### **1.0 Obtaining Certification**

##### **1.1 Certification Application Process**

Lab Management, Project Management, Sales & Marketing, and the QA Manager may initiate requests for certification or accreditation. The application is obtained, reviewed, and completed by the QA Manager or designee. Sections of the application may be

distributed as appropriate to various staff members to assist in completion.

The certifying agency's regulations should be carefully reviewed at the time of application to ensure any non-routine requirements are communicated to the laboratory.

The QA Manager consults with Lab Management, Project Management, and the Sales & Marketing Staff to determine if additional methods should be added to the current laboratory certification in order to support existing and future work as the laboratory's capabilities change.

## 1.2 Reciprocity

Reciprocity is a means of acknowledging another agency's certification via mutual agreements between certifying agencies. Many certifying agencies offer some type of reciprocal certification. The most common types of reciprocity are based on either TNI (NELAC) certification or homestate certification.

Homestate reciprocity refers to another state's certifying agency allowing a laboratory to perform work in that state, provided that the laboratory maintains accreditation within the state in which it resides.

TNI refers to The NELAP Institute which governs the NELAP Standard document outlining Quality System and laboratory functions and requirements. NELAP refers to the National Environmental Laboratory Accreditation Program. Many states will acknowledge a laboratory's TNI accreditation from another state.

Note: Reciprocal agreements between states do not afford a "blanket" certification. To obtain reciprocal certification, a laboratory must still apply for accreditation, submit all required application materials, and receive notification of certification – usually in the form of a certificate - from the reciprocal agency.

## 1.3 Records Maintenance

A copy of the original application, certificate, and related materials are maintained in accordance with SA-QA-001: *Document Control Program*. Copies of current certifications are kept in the Certifications folder on the public G-drive, which is accessible to all laboratory staff. In addition, copies of current laboratory certifications from Savannah and other TestAmerica facilities are maintained in the Proposal Library on the TestAmerica Oasis website and in the TotalAccess marketing tool. These documents may be required to support subcontracting and marketing activities.

## 1.4 Maintaining Certification

Most states require continued evidence of an effective Quality System in order for a laboratory to maintain certification. In addition to annual renewal applications, laboratories are often required to complete bi-annual PT studies with acceptable results obtained for each certifiable matrix/method/analyte combination. Annual MDL and continued demonstrations of analyst capability are also routinely required, in addition to on-site assessments.



## 1.5 Certification Tools and Records

There are several tools in place to aid laboratory staff in determining what certifications the laboratory maintains and understanding any state-specific analytical and/or reporting requirements.

### 1.5.1 Total Access

Total Access is a tool that can aid in determining which certifications the laboratory maintains. This tool is useful in the pre-project planning process.

### 1.5.2 State and Project Requirement Summaries

Some states and/or projects have specific analytical and/or reporting requirements. A summary of these requirements is kept in the State and Project Requirement Summary on the Public-QA Drive. These requirements must be reviewed by project management and laboratory staff prior to initiating work. The Project Manager must clearly note in the TALS Worksheet Notes and/or Project Plan if the Project Requirement Summary (PRS) is to be followed.

## 1.6 Information Resources

### 1.6.1 Agency Information

The QA staff maintains a controlled access database that lists current contact information for the agency that oversees laboratory certification as well as the regulatory programs that are offered for certification by the agency. This information may be provided as a resource to Lab Management, Project Management, Corporate QA, and the Sales & Marketing staff.

### 1.6.2 Certification Matrix

The QA Department ensures that the certification matrix maintained on Total Access is current. The QA Department ensures that the Certification Summary in the Proposal Library on the TestAmerica Oasis website is kept current.

### 1.6.3 Certification / Accreditation Maintenance Requirements

Laboratory Management is responsible for ensuring that laboratory operations are in compliance with the regulatory and certification program requirements for the jurisdiction in which certification is maintained.

The QA Department is responsible for maintaining up to date applications and program information including program specific regulations and requirements.

Project Management is responsible for verifying certification of analytes and methods requested by the client prior to accepting work and should be familiar with the state-specific requirements of that state.

## 1.7 Certifications Listing

At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Authority	Certification Number or Laboratory ID Number
A2LA (DoD ELAP)	0399-01
A2LA (ISO/IEC 17025)	399.01
Alabama	41450
Arkansas	88-0692
California	3217CA
Colorado	N/A
Connecticut	PH-0161
Florida	E87052
Georgia	803
Georgia EPD	N/A
Guam	09-005r
Hawaii	N/A
Illinois	200022
Indiana	N/A
Iowa	353
Kentucky	90084
Kentucky UST	18
Louisiana	30690
Louisiana	LA100015
Maine	GA00006
Maryland	250
Massachusetts	M-GA006
Michigan	9925
Mississippi	N/A
Montana	CERT0081

Authority	Certification Number or Laboratory ID Number
Nebraska	TestAmerica-Savannah
New Jersey	GA769
New Mexico	N/A
New York	10842
North Carolina DENR	269
North Carolina PHL	13701
Oklahoma	9984
Pennsylvania	68-00474
Puerto Rico	GA00006
South Carolina	98001
Tennessee	TN02961
Texas	T104704185-08-TX
USDA	SAV 3-04
Virginia	302
Washington	C1794
West Virginia DEP	94
West Virginia DHHR (DW)	9950C
Wisconsin	999819810
Wyoming	8TMS-Q

The certificates and accredited parameter lists are available for each State/Program at [www.testamericainc.com](http://www.testamericainc.com) under Analytical Services Search – Certifications. Copies of these document can also be found on the laboratory's public server, in TotalAccess, and in the QA offices.

## VOLATILE COMPOUNDS BY GC/MS


(Methods: EPA 624, EPA 8260B, and EPA 8260C)

### Approvals (Signature/Date):

 \_\_\_\_\_ November 3, 2014  
Andrea Teal  
Quality Assurance Manager

 \_\_\_\_\_ October 27, 2014  
Whitney Palefsky  
Environmental Health and Safety Coordinator

 \_\_\_\_\_ October 27, 2014  
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 \_\_\_\_\_ October 30, 2014  
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Facility Distribution No. 1

Distributed To: QA Navigator

## 1.0 **Scope and Application**

This SOP contains the procedures for the determination of volatile organic compounds (VOC) by purge and trap gas chromatography/mass spectrometry (GC/MS). This procedure is applicable to a wide variety of low molecular weight compounds that have low aqueous solubility and boiling points less than 200°C.

The routine matrices for this procedure are waters (EPA 624, EPA 8260B, and EPA 8260C) and soils (for EPA 8260B and EPA 8260C); however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1. This procedure may also be used to perform Custer Rule analyses for chloroform only (via a modified EPA 624 analysis).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

## 2.0 **Summary of Method**

Volatile organic compounds (VOCs) are purged from the sample matrix with nitrogen. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Water samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default. The calibration standards and the associated QC must be analyzed under the same conditions and volume. This sample introduction procedure is based on EPA 5030B, EPA 5030C, and EPA 624.

Low-level (nominally <1mg/kg) soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions. This sample introduction procedure is based on EPA 5030A (for soil samples collected in bulk) and EPA 5035A (for soil samples collected via Encore/Terracore devices).

High level soils (nominally >1mg/kg) and waste samples are extracted with methanol (1mL of methanol per gram of sample). An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

This SOP is based on EPA 624, EPA 8260B, and EPA 8260C.

### 3.0 **Definitions**

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

### 4.0 **Interferences**

#### 4.1 **Procedural Interferences**

- 4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.
- 4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions (found in Attachment 13) to ensure it is free from contaminants and does not contribute artifacts.
- 4.1.3 High purity reagents and solvents are used to help minimize interference problems. Methanol and Hydrochloric Acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.
- 4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.
- 4.1.5 VOCs commonly used in the laboratory are potential sources of contamination. Methylene chloride, acetone, Freon-113, MEK, hexane, toluene, and isopropanol are used in the laboratory and tend to present the most problems.
- 4.1.5 The Teflon seals of the purge and trap device can absorb and outgas many of the compounds that are included in this method. These Teflon fittings should be periodically checked for integrity. If the contamination is suspected, the fittings may be heated at 105°C for one hour or replaced.
- 4.1.6 The addition of acid to the sample during collection will cause the degradation of several target compounds. Acrolein and acrylonitrile recovery may be reduced and 2-chloro-ethyl vinyl ether will be completely degraded. The recovery of 2-chloroethyl vinyl ether will also be reduced as the purge and trap lines become acidic. For this reason, unpreserved vials must be utilized if these analytes are requested.

#### 4.2 **Matrix Interferences**

- 4.2.1 Matrix interferences may be caused by contaminants that are purged from the sample matrix.

Contamination by carryover can occur whenever high concentration samples and low concentration samples are analyzed sequentially. As such, samples known to be clean should be analyzed first. When practical, high concentration samples should be followed by a blank to check for cross-contamination. If the targets found in the highly

concentrated sample are found in subsequent samples, the analyst must verify that the P/T systems are not contributing contamination to the subsequent samples. If the target compound(s) are not present in subsequent samples, the analysis of a blank is not required but may be a prudent preventative measure. Frequent trap bakeout and purging of the entire purging system may be necessary when carry-over is suspected. Reagent blanks must be analyzed when contamination is suspected to ensure that the system is free from contamination.

A common type of contamination is from samples containing high concentrations of hydrocarbons such as gasoline or mineral spirits. At high concentrations, these compounds may cause elevated baselines that can obscure the mass ion signals of target compounds with masses similar to the hydrocarbons. For example, a common mass in hydrocarbons is mass 43, which is also present in many ketones. Mineral spirits at high concentrations can be very problematic as it contains hydrocarbons beyond C12, which can linger in the purge and trap unit and carryover for quite a few samples. High concentrations of hydrocarbons can also degrade the trap much more quickly than would be expected.

- 4.2.3 The volatiles laboratory must be kept as free from contamination as possible. Highly contaminated samples must be segregated from routine samples. Contact with sections of the laboratory where solvents are used should be minimized. Refrigerator and freezer blanks must be prepared, stored, and analyzed to evaluate the sample storage areas for possible contamination. Guidance is provided in SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*.
- 4.2.4 Matrix interferences may be overcome by the use of the secondary ions for quantitation. An example of this is the use of mass 82 for quantitation with chlorobenzene-d5 internal standard when a potential co-eluter, 1,1,1,2-tetrachloroethane, is a target compound. One of the mass fragments of 1,1,1,2-tetrachloroethane is mass 117, which is the recommended quantitation ion for chlorobenzene-d5. The use of the secondary ions should be used for quantitation in such cases when the laboratory can clearly demonstrate matrix problems. Mass 58 is recommended for quantitation of acetone due to the elution of a hydrocarbon at the same retention time.

## 5.0 **Safety**

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe,



nonabsorbent shoes when handling samples. Note: Cut-resistant gloves should be worn, or other hand protection material used, when opening and closing VOA vials.

#### 5.1 Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst must be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The exit vent of the split injector must have a carbon trap in-line to collect the volatile compounds that are vented during the injection of the sample. The traps should be changed a minimum of every three months and must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM.

Methanol is a flammable solvent. It can cause irritation to the respiratory tract. Overexposure can cause fatigue, confusion, headache, dizziness, and drowsiness.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should be used in a fully functional fume hood.

#### 5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their SDS/MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the SDS/MSDS for each material before using it for the first time or when there are major changes to the SDS/MSDS. Electronic copies of SDS/MSDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit <sup>1</sup>	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200ppm TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Hydrochloric Acid	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
<sup>1</sup> Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

## 6.0 **Equipment and Supplies**

### 6.1 **Equipment and Instrumentation**

A list of the instruments, with their basic configuration, is provided in Attachment 9. These instruments were in use at the time of SOP revision. Other instruments and configurations may be used provided they are fully documented and validated in accordance with laboratory procedures.

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

### 6.2 **Analytical Data System / Software / Hardware**

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's TALS system. CHROM software has the capability of processing stored GC/MS data by recognizing a GC peak within any given retention time window, comparing the mass spectrum from the GC peak with spectral data in a user-created data base, and generating a list of tentatively identified compounds with their retention times and scan numbers. The software also allows integration of the ion abundance of any specific ion between specified time or scan number limits, calculation of response factors as or construction of a linear regression calibration curve, calculation of

response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

### 6.3 Lab Supplies

Supelco Vocab 3000 trap or equivalent – Other traps may be used as long as the target compounds can be detected at the required quantitation limit.

Recommended Column: J&W DB-624: 20m x 0.18mm ID, 1.8um film

### 6.4 Volumetric Containers and Dispensers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Standard Preparation	None (Purchased Class A)	None (Purchased Class A)
Gas-Tight Syringes	Various	QUANTITATIVE	Standard Preparation Spike Addition	None, if received with COA	None, if received with COA
Pump-Style Mechanical Pipettes	5mL	QUANTITATIVE	Reagent/Water Dispensing	Monthly (Daily for DOD)	Accuracy = 2% Precision = 1%
VOA Vials*	40mL	QUANTITATIVE	Sample Collection Instrument Analysis	Per Lot	Accuracy = 2% Precision = 1%

\*Note: The VOA vials used to collect samples have been verified to actually contain closer to 43mL. As such, spiking amounts for ISSU have been adjusted accordingly. Standards are prepared in 50mL volumetric containers, and poured into the VOA vial for analysis on the instrument.

### 6.5 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination. Containers are received from vendors with a statement that the containers are suitable for the intended use and have been tested and certified to be free of contamination.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the containers routinely used to collect field samples.

## 7.0 Reagents and Standards

### 7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent

materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it.

## 7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*.

Methanol and Hydrochloric Acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

7.2.1 Reagent water – ASTM Type II; lab generated water

7.2.2 Blank sand – Macro Chemicals; used as blank matrix for soil samples; Purify by heating at 160°C for four hours or longer in a shallow tray.

7.2.3 Methanol – for purge and trap analysis; J.T. Baker 9077-02 (1L)

## 7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*. Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

The recipes for the preparation of standards are provided in Attachment 10. The recipes contain the stock standards, preparation steps, storage, and expiration dates for the routine target compounds.

## 8.0 Sample Collection, Preservation, and Handling

Refer to Attachment 2 for a summary of the routine containers, holding times, preservation requirements, etc.

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for information on the preservation and dechlorination checks required for these methods.

## 9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the QC Summary in Attachment 3 provide requirements for evaluating QC data.

### 9.1 Batch QC

All batch QC must meet the criteria given in Attachment 3 of this SOP.

#### 9.1.1 Soil Samples

A batch consists of up to 20 environmental samples and the associated QC items. The laboratory defaults to the following as the minimum QC items required for each batch: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

If there is insufficient sample to perform the MS/MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample is defined as receiving less than 30g for bulk samples or less than 4 Encores/Terracores.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

#### 9.1.2 Aqueous Samples

A batch consists of up to 20 environmental samples and the associated QC items. The minimum QC items required for each batch are as follows:

EPA 8260B and EPA 8260C:  
method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

EPA 624:  
method blank, a laboratory control sample (LCS), a matrix spike (MS) performed per 10% of samples analyzed, and a matrix spike duplicate (MSD). This equates to 1 MS and 1MSD for a batch of 10 or less samples or equates to 1 MS (from sample 1-10), 1 MS (from sample 11-20), and 1 MSD for a batch of 11-20 samples.

If there is insufficient sample to perform the required MS and/or MSD, the LCS must be prepared in duplicate (i.e., LCS/LCSD). An NCM must be initiated on all affected samples to denote when insufficient sample is provided to perform a MS/MSD. Insufficient sample is defined as receiving less than less than 4 vials.

Note: It is not uncommon to for the laboratory to be supplied with insufficient containers to perform MS/MSD; therefore, the laboratory routinely performs LCS/LCSD as a default. If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

#### 9.1.3 Poor Performers / Erratic Compounds

As indicated in EPA 8260B and EPA 8260C and/or via assessment of laboratory control sample recoveries and control charts, the compounds listed in Attachment 11 are Poor Performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count, provided their %R is >10%.

Note: An NCM must be initiated to denote this situation.

## 9.2 Instrument QC

The term “clock time” or “analytical clock” refers to the amount of time that can pass before additional instrument QC items must be performed. The analytical clock begins with the injection of the BFB, and all subsequent injections must be completed before the clock time expires - at which point new instrument QC is performed and a new clock is initiated.

The clock times are defined as follows:

- EPA 8260B and EPA 8260C = 12 hours
- EPA 624 = 24 hours
- EPA 624 Cluster Rule (chloroform only) = 8 hours

Note: Due to instrument configurations employing dual concentrators, most of the laboratory instruments can analyze more than 20 injections within the designated clock times. An analytical batch is still defined as 20 field samples; therefore, if more than 20 field samples are analyzed within a clock, additional batch QC is required (i.e., another method blank, LCS, and LCSD or MS/MSD must be performed).

### 9.2.1 BFB Tune Check

9.2.1.1 Fifty nanograms of 4-BFB must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. Meeting the tuning criteria ensures that the instrument is measuring the proper masses in the proper ratios. The 4-BFB analysis takes place under the same instrument conditions as the calibration standards and samples except that a different temperature program can be used to allow for the timely elution of 4-BFB. All other instrument conditions must be identical - the mass range, scan rate, and multiplier voltage.

If the instrument is configured for direct injection, 50ng of 4-BFB may be injected directly on to the column. If the purge and trap is used to analyze the 4-BFB, the purge and trap conditions must be the same as for the calibration standards and samples.

### 9.2.1.2 Evaluation of the 4-BFB peak

- 9.2.1.2.1 The chromatogram must exhibit acceptable baseline behavior and the 4-BFB peak must be symmetrical (i.e., Gaussian). A spectrum of the baseline that shows high abundances of mass 40 (Argon) and mass 44 (carbon dioxide) may indicate a leak or contaminated carrier gas.
- 9.2.1.2.2 The spectrum of the 4-BFB must meet the criteria listed in Attachment 7. Background subtraction must be straightforward and designed only to eliminate column bleed or instrumental background. Scans  $\pm 1$  scan from the apex can be evaluated for the 4-BFB criteria. Consecutive scans within this range can be averaged to meet the criteria.
- 9.2.1.2.3 The 4-BFB analysis should be evaluated as to the relative size of the 4-BFB peak under the m/z 95 profile. A benchmark area window should be established for each instrument. Response outside of this window suggests instrumental



problems such as a poor purge, clogged jet separator, leak in the Tekmar purging device, reduced or elevated detector sensitivity, improper electron multiplier voltage selection, wrong tune method or tune file selected for this analysis, PFTBA valve left open, or other anomalies.

- 9.2.1.2.4 If the 4-BFB fails to meet the acceptance criteria, the instrument may require tuning (manually or automatically with PFTBA). Depending on the nature of the results from the 4-BFB analysis, other corrective measures may include remaking the 4-BFB standard and/or cleaning the mass spectrometer source.

## 9.2.2 Trap Check Standard

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds.

- 9.2.2.1 Prepare the trap check standard by injecting 2 $\mu$ L of a 50 $\mu$ g/mL bromoform standard into 5mL of reagent water. Other sample volumes may be used but the sample must transfer 100ng of bromoform to the column. Add the internal standards and surrogates. Analyze the sample using the same analytical system conditions used for samples and standards.

- 9.2.2.2 Evaluate the chromatogram for the presence of chloromethane and bromomethane. Compare the response to the 1.0 $\mu$ g/L standard. The response should be less than or equal to the reporting limit.

Note: Ensure sure that the spectra match the reference spectra and that the most abundant ions are present for both compounds - chloromethane ( $m/z$  50, 52) and bromomethane (94, 96).

- 9.2.2.3 If the trap check standard does not meet the acceptance criteria, the trap must be replaced and conditioned.

## 9.2.3 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

The initial calibration standard concentrations currently in use in the laboratory are as follows:

EPA 8260B and EPA 8260C:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)	Final Concentration – Soils (ug/kg)
1	5	1.0	
2	25	5.0	5.0
3	50	10	10
4	100	20	20
5	250	50*	50*
6	500	100	100
7	1000	200	200

\*CCV Level

EPA 624:

Standard Level	Concentration (ng)	Final Concentration – Waters (ug/L)
1	5	1.0
2	25	5.0
3	50	10
4	100	20*
5	500	100
6	1000	200

\*CCV Level

Refer to Attachment 10 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

### 9.2.3.2 ICAL Criteria

#### 9.2.3.2.1 EPA 8260B

The initial calibration is evaluated specifically for the calibration check compounds (CCC) and the system performance check compounds (SPCC). The CCC and SPCC criteria are given in Attachment 5 of this SOP. The %RSD criteria for CCC and minimum RRF for SPCC must be met before the analysis of samples can begin.

After the CCC and SPCC initial calibration criteria have been met, each target must be evaluated for linearity. The relative standard deviation of the calibration standards must be <15% for the initial calibration curve to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. The regression coefficient ( $r^2$ ) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

#### 9.2.3.2.2 EPA 8260C

The relative standard deviation (RSD) should be less than 20% difference. If a compound exceeds 20% RSD, a regression curve should be employed. The regression coefficient ( $r^2$ ) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable. A list of compounds with minimum relative response factor criteria is listed in Attachment 6.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted. Some programs and agencies (e.g., SC DHEC) do not allow the use of quadratic curves. Refer to the Project Requirement Summary and/or Project Plan to determine if this curve type is prohibited.

The method of linear regression has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods do not have this potential bias. It is preferred to use a weighted linear fit. Additionally, the lowest calibration point should be within 30% of the standards true concentration. Analytes that do not meet the minimum quantitation calibration refitting criteria should be considered "out of control" and corrective action should be taken such as re-evaluating the lower quantitation limit or reporting the "out of control" analytes as estimated when the concentration is at or near the lowest calibration point.

Note: 10% of the analytes in the calibration may be outside criteria. If analytes do meet criteria, they are considered estimated. An NCM must be initiated to denote analytes that do not meet the RSD or  $r^2$  criteria.

Note: For any analyte associated with a calibration or minimum response factor, an RLCCV must be analyzed with each subsequent clock. An NCM must be initiated to denote this situation. Any positive results should be noted as estimated.

#### 9.2.3.2.3 EPA 624

The relative standard deviation of the calibration standards must be <35% for the initial calibration curve to be acceptable. If one or more compounds do not meet the %RSD criterion of <35%, the next option is to evaluate a regression curve. The regression coefficient ( $r^2$ ) of the regression curve must be greater than 0.990 for the initial calibration curve to be acceptable.

#### 9.2.4 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to the CCV concentration. Refer to Attachment 10 for the standard preparation

instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

#### 9.2.4.1 EPA 8260B ICV Criteria

All compounds must be within 30%D for the ICV to be acceptable.

The analytes listed in Attachment 11 behave erratically and/or are poor performers; therefore, the acceptance criteria for these analytes are 60%D.

#### 9.2.4.2 EPA 8260C ICV Criteria

All compounds must be within 30%D for the ICV to be acceptable. Analysis may continue for those analytes that fail the criteria with the understanding that the results be flagged as estimated.

The analytes listed in Attachment 11 behave erratically and/or are poor performers; therefore, the acceptance criteria for these analytes are 50%D.

#### 9.2.4.3 EPA 624 ICV Criteria

The initial calibration verification (ICV) is acceptable if each analyte meets the criteria outlined in Attachment 12.

#### 9.2.5 Continuing Calibration Verification (CCV)

The initial calibration curve must be verified at the beginning of each clock with a mid-level standard.

The continuing calibration verification standard concentration currently in use in the laboratory is specified in Section 9.2.3. Refer to Attachment 10 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

##### 9.2.5.1 EPA 8260B CCV Criteria

For EPA 8260B, the CCC and SPCC criteria (Attachment 5) must be met for the CCV to be acceptable. For non-CCC and non-SPCC compounds, %D must be within 20% for the CCV to be acceptable.

The analytes listed in Attachment 11 behave erratically and/or are poor performers; therefore, the acceptance criteria for these analytes are 60%D.

Note: The SPCC criteria must be met even if the regression curve option is used for quantitation.

In addition to the response criteria given in this section, the CCV must be evaluated for internal standard response. The extracted ion current profile (EICP) area for each of the internal standards in the CCV must be within -50% to +100% from the last initial calibration sequence to be acceptable. If these criteria are not met, the analytical system must be inspected for problems and corrective action instituted.

#### 9.2.5.2 EPA 8260C Criteria

For EPA 8260C, the minimum response factor criteria (Attachment 6) of the initial calibration must be met.

All compounds should be within 20% of the true value (%difference for average or %drift for regression fits). Due to the large number of compounds being analyzed by this method some compounds may fail to meet this criteria. The CCV is deemed to be acceptable as long as no more than 20% of the compounds exceed this amount and adequate sensitivity with analysis of a standard (a standard at or below the reporting limit) can be demonstrated to report these compounds as non-detects. If one of the failed compounds is present, the concentration must be reported as an estimated value.

An NCM must be initialed to denote situation.

The analytes listed in Attachment 11 behave erratically and/or are poor performers; therefore, these analytes may exceed the CCV %D criteria such that their %D may be >20% if the average %D of all the analytes in the ICV is <30%. An NCM must be initiated to denote this situation.

#### 9.2.5.3 EPA 624 CCV Criteria

For EPA Method 624, all target analytes must meet the criteria outlined in Attachment 12 to be acceptable.

#### 9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Fluorobenzene, 1,4-dichlorobenzene-d4, and chlorobenzene-d5 are the internal standards.

Prior to analysis, the internal standards must be added to all standards, samples, and QC items. The concentration of the internal standards must be the same in all calibration samples, field samples, and QC samples. A concentration of 50ug/L is used for each of the ISTD analytes.

The response of the ISTD in the ICV/CCV must be within -50% to +100% of the response of the ISTD in the CCV-level standard in the initial calibration sequence. If the response is outside of this range, the analysis of the CCV must be repeated and any samples associated with the CCV must also be re-analyzed. Repeated failure of the ISTD response will require re-calibration.

The response of the ISTD in the samples and batch QC items must be within -50% to +100% of the response of the previous CCV. If the response is outside of this range, corrective action must be taken.

#### 9.2.7 Surrogates

This procedure uses surrogates to evaluate the analytical process. Dibromofluoromethane, 1,2-dichloroethane-d4, toluene-d8, and p-BFB are the surrogates.

Prior to preparation, these surrogates must be added to all samples and QC items. The concentration of the surrogates must be the same in all field samples and QC samples. A concentration of 50ug/L is used for each of the surrogate analytes.

The percent recovery of the surrogate in all field samples and QC samples must be within the limits listed in the Method Limit Groups (MLGs) in TALS. If the percent recovery is outside of this range, the analysis of the sample must be repeated. Repeated failure of the surrogate percent recovery may indicate re-extraction is necessary.

### 9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

## 10.0 Procedure

### 10.1 Preparation

#### 10.1.1 Aqueous Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

Using a 50uL syringe, add 43uL of the ISSU to the sample, injecting the solution through the vial septum. Invert the vial several times to mix. Transfer to the instrument.

#### 10.1.2 Aqueous QC Preparation

The method blank and LCS are prepared in reagent water using a 50mL volumetric flask.

- For the method blank, add 50uL ISSU to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the LCS, add 50uL ISTD, 50uL Mega Mix, and 150uL MeOH to 50mL reagent water. Pour into a VOA vial and place on the instrument.
- For the MS/MSD, add 43uL ISTD, 43uL Mega Mix, and 129uL MeOH into each sample vial selected for the MS and MSD. Place on the instrument.

#### 10.1.3 Soil Sample Preparation

Refer to SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples* for the soil specific sample preparation procedures. This information is summarized below.

Three Terracore/Encore devices and one bulk container are routinely received for each soil sample. Two of the Terracores/Encores are prepared for low level analysis and one is extracted in methanol for medium-level analysis to be used if



the low-level samples exceed the calibration range. The bulk container is used for determining the type of preservation for the low-level samples and for screening.

At the time of analysis, the following steps are taken:

For low-level samples, 5mL reagent water and 5uL ISSU are added directly to the samples vial. The sample is then ready for analysis.

For medium-level samples, a dilution is prepared and analyzed. The default dilution is a DF40 (i.e., using 125uL of sample). 5uL VOA Surrogate Mix is added directly to the sample vial (if 5mL of MeOH were used) or 10uL VOA Surrogate Mix is added (if 10mL of MeOH were used). 5uL ISTD is added. The sample is then ready for analysis.

#### 10.1.4 Soil QC Preparation

For low-level samples, the method blank and LCS are prepared using blank sand directly in a VOA vial.

- For the method blank, add 5g sand, a stir bar, 10mL reagent water, and 5uL ISSU.
- For the LCS, add 5g sand, a stir bar, 10mL reagent water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol.
- For the MS/MSD, add 5mL water, 5uL ISTD, 5uL Mega Mix, and 15uL methanol directly to the selected sample.

For medium-level samples, the method blank and LCS are prepared using Ottawa sand directly in a VOA vial.

- For the method blank, add 5g blank sand, 5mL methanol, and 5uL VOA Surrogate Mix. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.
- For the LCS, add 5g blank sand, 5mL methanol and 250uL Mega Mix. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.
- For the MS/MSD, add 5g blank sand, 5mL methanol and 250uL Mega Mix to the sample containers selected for the MS/MSD. Remove a 125uL aliquot and add to 10mL reagent water spiked with 5uL ISTD.

## 10.2 Analysis

### 10.2.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The purge time must be 11 minutes. All other parameters are optimized by the lab for the target compounds and documented in the maintenance log. Therefore, the actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

#### GC Parameters

Column: 20m x 0.18mm ID x 1.0um  
Column: Flow: 0.5-1.0mL/min  
Injector: Split/Splitless operated in the split mode with 1mm ID quartz insert  
Split ratio (desorb to column flow): 40:1 to 80:1

#### MS Parameters

Mass spectrometer interface: 240°C (direct column interface)  
Mass spectrometer source temperature: 250°C  
Mass scan range: 35-300amu, with a minimum scan cycle of 1 scan per second  
Injector: 250°C

Temperature Program:  
Initial Temperature: 50°C  
Program Rate: 18°C/minute  
Final Temperature: 200°C

#### Example Purge and Trap Conditions

The purge and trap conditions listed in this section are for guidance. The lab must document the actual conditions used. The purge time must be 11 minutes. Other parameters may be varied to optimize the detection of the target compounds.

VOCARB 3000 trap  
Purge Time: 11 minutes  
Purge temperature: aqueous-ambient; soils-heated 40°C  
Desorb time: 0.50 minutes  
Desorb temperature: 250°C  
Bake time: 4 to 6 minutes at 260°C  
Purge flow: Approximately 30-40mL/minute  
Valve temperature: 150°C  
Transfer line: 150°C

The purge flow must be balanced for adequate sensitivity of the target compounds. If the purge flow is too high, the response of the gases will be low and not reproducible. The SPCC criteria for chloromethane may not be achieved if the purge flow is too high. If the purge flow is too low, the response of the more water-soluble targets - ketones, ethers, bromoform - may be low and the reporting limit may not be achieved on a routine basis.

#### 10.2.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. Instructions for the addition of the internal standard spiking mix are given in the previous sections.

#### 10.2.3 BFB Tune Check

A BFB tune check must be analyzed at the beginning of each clock as a check on the "tune" of the mass spectrometer. This check must meet the criteria described given in Section 9.2.1.

If the instrument is configured for direct injection, inject 2uL of the 25ng/uL solution into the GC and analyze the 4-BFB using the instrument conditions listed in Section 10.1. A solvent delay should be set to allow the methanol to pass through the mass spectrometer while the MS is "off".

If the purge and trap is used to introduce the 4-BFB into the MS, add 1uL of the 50ng/uL solution to 5.0mL of reagent water. Transfer the 5.0mL to the purge and trap device and purge the sample using the same conditions used for sample analysis.

#### 10.2.4 Trap Check Standard

The trap check standard is used to evaluate the condition of the trap by monitoring the formation of chloromethane and bromomethane. Chloromethane and bromomethane may be formed on a degraded trap by thermal decomposition of halogenated compounds. This check must meet the criteria described given in Section 9.2.2.

#### 10.2.5 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.3. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.4, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.6.

#### 10.2.6 Sample Analysis

The sample/extract must be injected using the same injection volume used for the calibration standards. The samples can only be analyzed after the tune, the trap check, the calibration (initial or continuing), and the method blank and LCS criteria have been met. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

#### 10.2.7 Example Analytical Sequence

A typical example analytical sequence is listed below.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch	

QC Items	
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	

## 11.0 Calculations / Data Reduction

### 11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data*. This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data review and reporting must be performed in accordance with SA-QA-002: *Data Generation and Review*.

#### 11.1.1 Target Analyte Identification

A target compound is identified by the visual comparison of the sample mass spectrum with the mass spectrum of the target compound from a reference spectrum of the target compound stored in a library generated on the same instrument or a standard spectral library such as the NIST/NBS.

##### 11.1.1.1 Two criteria must be met in order to identify a target compound:

- Elution of the sample component within  $\pm 0.06$  Relative Retention Time (RRT) units of the daily standard containing that compound. The RRT is calculated as follows:

$$RRT = \frac{\text{retention time of the target compound}}{\text{retention time of the associated internal standard}}$$

- Correspondence of the target compound spectrum and the standard component mass spectrum

##### 11.1.1.2 All ions present in the standard component mass spectrum at a relative intensity greater than 10% (most abundant ion = 100%) should be present in the sample component mass spectrum. Other ions may be present in the sample component. Coelution of a non-target compound with a target compound will make the identification of the target compound more difficult. The ions due to the non-target compound should be subtracted from the sample component spectrum as part of the background to account for the discrepancy between the sample spectrum and the standard spectrum.

- 11.1.1.3 The relative intensities of the ions present in the sample component spectrum should agree within  $\pm 30\%$  of the relative intensities of the ions in the standard reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum should have a corresponding abundance between 20% and 80% in the sample component spectrum.
- 11.1.1.4 If the above criteria are not met exactly, the analyst should seek help from a senior analyst or the Department Manager. If there is sufficient evidence to support the identification of the component, then the component is identified, quantified, and reported.

#### 11.1.2 Dilutions

Samples may be screened prior to analysis as outlined in SOP SA-VO-001. This screening data provides qualitative information that is used to determine if dilutions are required for analysis.

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

#### 11.1.3 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

#### 11.1.4 Chemical Relationships

Benzene, toluene, ethylbenzene, and the xylenes are generally present together in samples and indicate the presence of gasoline

m/p-Xylenes are generally higher than o-xylene

Hydrocarbons present in samples containing gasoline generally contain mass 43 and may co-elute with target analytes with mass 43 as the quant or confirmation ion or may skew the spectrum of a compound with mass 43 as part of the spectrum.

Cis-isomers are generally more prevalent than the trans- isomers

Pay particular attention to the retention time of isomer because the only way to positively identify them is by retention time. The isomers are:

1,1-dichloroethane and 1,2-dichloroethane  
1,1-dichloroethene, cis-1,2-dichloroethene, and trans-1,2-dichloroethene  
1,1,1-trichloroethane and 1,1,2-trichloroethane  
ethyl benzene, m/p-xylene, and o-xylene  
1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene  
1,1-dichloropropene, cis-1,2-dichloropropene, and trans-1,2-dichloropropene  
2-chlorotoluene and 4-chlorotoluene  
1,2,3-trichlorobenzene and 1,2,4-trichlorobenzene  
1,3,5-trimethylbenzene and 1,2,4-trimethylbenzene  
4-methyl-2-pentanone (MIBK) and 2-hexanone  
n-butylbenzene, sec-butylbenzene, tert-butylbenzene, and isopropylbenzene

Higher chlorinated alkanes and alkenes may have lower chlorinated alkanes or alkenes present due to degradation. The following table lists some common chlorinated compounds and their degradation products. Look for the degradation product(s) when the concentration of the compound in the left column is present at high concentrations.

Analyte	Degradation Product
1,1,2,2-tetrachloroethane	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) vinyl chloride 1,1,2-trichloroethane (1,1,2-TCA) 1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,2-trichloroethane (1,1,2-TCA)	1,2-dichloroethane (1,2-DCA) Chloroethane
1,1,1-trichloroethane (1,1,1-TCA)	1,1-dichloroethene (1,1-DCE) 1,1-dichloroethane (1,1-DCA) Chloroethane
Carbon tetrachloride	Chloroform Methylene chloride Chloromethane
Tetrachloroethene (PCE) (PCE = perchloroethylene which is a common name for tetrachloroethene)	trichloroethene (TCE) cis-1,2-dichloroethene (c-1,2-DCE) trans-1,2-dichloroethene (t-1,2-DCE) Chloroethene
1,2,4-trichlorobenzene	1,4-dichlorobenzene (1,4-DCB) 1,2-dichlorobenzene (1,2-DCB) Chlorobenzene

## 11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.



11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

CONC<sub>Sample</sub> = Concentration of the sample extract (at the instrument)

F = Final volume of the extract

I = Initial volume/weight

dw = % Solids decimal equivalent

D = Dilution factor

Note: All dry weight corrections are performed automatically in TALS.

Note: This formula assumes all unit conversion factors are applied.

Note: Procedures for entering soil preparation and dilution data into TALS and CHROM are given in Attachment 14 of this SOP.

## 12.0 **Method Performance**

### 12.1 **Reporting Limit Verification (RLV)**

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

### 12.2 **Method Detection Limit (MDL) Study**

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

### 12.3 **Method Detection Limit Verification (MDLV)**

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

## 12.4 QC Limit Generation, Control Charting, and Trend Analysis

### 12.4.1 EPA 624

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

### 12.4.2 EPA 8260B and EPA 8260C

The control limits for the batch QC items (LCS/LCSD and MS/MSD) for this procedure are not specified by the reference method; therefore, the laboratory defaults to in-house and/or laboratory-derived limits for the evaluation of batch QC items.

Control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

## 12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability (IDOC). The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or by the analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

## 12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

### **13.0 Pollution Control**

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

### **14.0 Waste Management**

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

#### **14.1 Waste Streams Produced by the Method**

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah Addendum to the EHSM.
- Methanolic waste from rinsings and standards – Transfer to satellite container for methanolic (flammable) waste. Transfer to hazardous waste section when satellite container is full.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.

### **15.0 References / Cross-References**

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006: *Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- SOP SA-VO-001: *Preparation, Screening, and Storage of Volatiles Samples*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Test Methods for Evaluating Solid Wastes, SW-846; Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, DC.
  - Method 5030B
  - Method 5030C
  - Method 5035A
  - Method 8000B
  - Method 8000C
  - Method 8260B
  - Method 8260C
- EPA Method 624: *Purgeables*. 40 CFR Part 136, Appendix A, July 1, 1995.

## **16.0 Method Modifications and Clarifications**

### **16.1 Incorporation of Other Matrices**

This procedure may be modified to analyze other matrices (e.g., wipe, waste, and TCLP/SPLP leachate samples) based on the needs of the client. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe and waste matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate waste samples and its routine water RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate wipe samples. Soil and/or water DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices, as applicable. The laboratory uses its routine aqueous RLs (converted for initial and final volumes, etc.) and default QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Ottawa sand is used as the blank matrix for wipes and wastes unless specifically requested otherwise by the project.

#### **16.1.1 Collection and Handling Procedures**

Waste (oil) samples are routinely collected in 40mL VOA vials. Waste (oil) samples must be iced at the time of collection and maintained at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. Samples must be analyzed within 14 days of

collection.

Wipe samples are routinely collected in 40mL VOA vials. Wipe samples must be iced at the time of collection and maintained at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. Samples must be analyzed within 14 days of collection.

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a Tedlar bag. TCLP/SPLP leachates must be stored at 0-6°C (i.e., less than 6°C with no frozen samples) until time of preparation. The leachate sample must be analyzed within 14 days of completion of the TCLP/SPLP extraction.

#### 16.1.2 Preparation and Analytical Procedures

Wipe samples are analyzed in the same fashion as water samples. Refer to Work Instruction FQA088: *Wipe Tests: Sampling and Analysis* for additional information on wipe procedures.

Waste samples are prepared and analyzed in the same fashion as medium-level soil samples. Note: Waste samples often require large dilutions.

TCLP/SPLP matrices are prepared and analyzed in the same manner as routine water samples; however, a default dilution factor of 20 is used.

#### 16.2 Other Considerations

16.2.1 EPA Method 8260B does not contain calibration verification criteria for non-CCC analytes nor does it require non-CCC analytes to be evaluated for response; however, the laboratory has adopted in-house criteria for non-CCC analytes as outlined in this SOP.

16.2.2 EPA Method 8260B does not require the analysis of an ICV. NELAC requires an ICV; however, it does not list specific criteria. The laboratory has adopted in-house criteria for ICVs for EPA 8260B as outlined in this SOP. The laboratory satisfies this requirement via the ICV performed with each ICAL.

16.2.3 EPA Method 8260B allows alternate criteria to be used for the BFB evaluation. As such the laboratory has incorporated criteria the CLP OLMO4.0 (January 1998) method.

16.2.4 The laboratory has defined the analytes listed in Attachment 11 as poor or erratic performers and allows for exceptions to the ICV, CCV, LCS/LCSD, MS/MSD, and Sporadic Marginal Exceedance criteria for these analytes as outlined in this SOP.

16.2.5 It has been determined that increased methanol concentrations can suppress the response of the gas compounds, leading to erratic recovery and reduced linearity. As such, the laboratory normalizes the volume of methanol in all standards and QC items to minimize this effect. By introducing a constant volume of methanol to initial calibration standards, CCV/ICV, LCS, and MS/MSD, better sensitivity and recovery of these analytes is achieved.

16.2.6 The 7-day holding time for samples with pH>2 is listed in EPA 624 and is not included in the SW-846 methods. The laboratory has adopted this as internal guidance for EPA

8260B and EPA 8260C as is common industry practice and will make every effort to analyze samples with pH>2 within 7 days.

- 16.2.7 Historical standard practice for most laboratories was to combine all analytes into one analysis using a single acid preserved container. This practice is still acceptable in those cases where the compounds of interest are not adversely affected by the addition of the hydrochloric acid (HCl) preservative; however, EPA 624 and EPA 8260B both list special preservation requirements for acrolein, acrylonitrile, and 2-chloroethylvinyl ether (2-CEVE). Although these analytes are rarely found in the environment, preservation at pH<2 (as achieved using HCl preservative) breaks down these analytes and may result in a significantly low bias and/or non-detect value.

The reference methods suggest to preserve these analytes to a pH between 4-5. Achieving this narrow pH range is problematic; therefore, the only other alternative is to use an unpreserved container for these analytes. As such, if acrolein, acrylonitrile, and/or 2-CEVE are on the requested target analyte list, the laboratory now defaults to using an unpreserved container. The other target analytes reported for these methods are not adversely affected by using an unpreserved container provided they are analyzed within the shorter, method-prescribed holding times (HT) defined for unpreserved samples. Specific information on VOA holding times, preservation requirements, and analyte-specific requirements is given in Attachment 2.

- 16.2.8 The 40mL VOA vials used by the laboratory have been demonstrated to actually contain approximately 43mL. As such, standard spiking amounts have been adjusted accordingly to accommodate for this volume (e.g., spiking 43uL instead of 40uL).

- 16.2.10 The laboratory has incorporated the batch QC items as outlined in Section 9.1. Some additional QC items are performed which differ from those specified in the reference methods (e.g., LCSD or MSD) to satisfy common state regulatory requirements and/or client requests for precision data and/or to facilitate scheduling and data evaluation. The method-specified batch QC items are as follows:

EPA 8260B and EPA 8260C:  
Method blank, LCS, MS, and sample duplicate or MSD

EPA 624  
Method blank, LCS, MS per 10% of samples analyzed

- 16.2.11 EPA Method 624 specifies a required concentration range criteria for the evaluation of QC items (i.e., the Q-Table). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

- 16.2.12 The EPA 8260B reference method specifies a 1-week expiration date for the gas standards. The laboratory prepares a 25mL volume of MegaMix standard and splits it into five 5mL Mini-nert vials. Each vial is assigned a 1-week expiration date from the date that vial is opened, not to exceed 14 days from the preparation date of the 25mL volume. Note: This expiration date is a maximum. Shorter expiration dates may be used based on evidence of gas compound volatility as evaluated via CCVs.

- 16.2.13 Due to client requests, the analyte list utilized by the laboratory may be different than those listed in the reference methods.

**17.0 Attachments**

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

- Attachment 1: SOP Summary
- Attachment 2: Sample Collection, Preservation, and Holding Time Table
- Attachment 3: QC Summary
- Attachment 4: Preventative Maintenance and Troubleshooting
- Attachment 5: EPA 8260B Calibration Criteria: SPCCs and CCCs
- Attachment 6: EPA 8260C Minimum Response Factors
- Attachment 7: BFB Tune Criteria
- Attachment 8: Target Compound Information: Ions and ISTDs
- Attachment 9: Instrument Configurations
- Attachment 10: Standard Information and Recipes
- Attachment 11: Poor Performers
- Attachment 12: EPA 624 CCV & ICV Criteria (Q-Table)
- Attachment 13: Glassware Cleaning Procedures
- Attachment 14: Data Entry Procedure Work Instruction



## Attachment 1: SOP Summary

### Sample Preparation Summary

Aqueous samples are routinely purged at ambient conditions; however, a heated purge may be used if required by the project. A 5mL purge volume is used as the default. The calibration standards and the associated QC must be analyzed under the same conditions and volume.

Low-level soil samples are purged at 40°C in a purge and trap instrument designed to add water and internal standards to the vial containing the sample without breaking the seal. The sample is stirred during purging to thoroughly mix the soil and water. The calibration standards and associated QC are purged under the same conditions.

High level soils and waste samples are extracted with methanol (1mL of methanol per gram of sample). An aliquot of the methanol extract is injected into reagent water. The methanol extract/reagent water is purged at ambient temperature using the same instrument conditions and calibration used for aqueous samples.

### Sample Analysis Summary

VOCs are purged from the sample matrix with nitrogen. The VOCs are transferred from the sample matrix to the vapor phase. The vapor is swept through a sorbent tube where the VOCs are trapped. After the purging is completed, the trap is heated and backflushed with helium to desorb the VOCs onto a GC column. The GC is temperature-programmed to separate the VOCs, which are then detected by a mass spectrometer. Qualitative identification of the target compounds in the sample is based on the relative retention time and the mass spectra of the characteristic masses (ions) determined from standards analyzed on the same GC/MS under the same conditions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

Description	Comments
Blank	
BFB	Clock time begins with injection of the BFB
Initial Calibration	
ICV	Second Source
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	
BFB	Clock time begins with injection of the BFB
CCV	
LCS	
LCSD	Required if insufficient sample is provided for MS/MSD
Trap Check	
MB	
Samples & Batch QC Items	

## Attachment 2: Sample Collection, Preservation, and Holding Time Table

Aqueous samples are routinely collected in 40mL VOA vials. The table below outlines the laboratory's default procedures for aqueous samples:

Method	Acrolein, Acrylonitrile, or 2-CEVE Requested?	Chemical Preservation	Thermal Preservation	Holding Time
EPA 8260B EPA 8260C	Yes	None	0-6°C	7 days
EPA 8260B EPA 8260C	No	HCl (pH<2)	0-6°C	14 days
EPA 624	Yes	None	0-6°C	3 days*
EPA 624	No	HCl (pH<2)	0-6°C	14 days

\*Note: 3-day HT is specific to acrolein. If acrolein is not requested, 7 days is used for the remaining target compounds.

The table below outlines the laboratory's procedures for soil samples:

Sample Container	Chemical Preservation	Thermal Preservation	Holding Time
Terracore Kit: 5g Terracore sampler, 2 x pre-weighed 40-mL VOA w/ H <sub>2</sub> O, pre-weighed 40-mL VOA w/ MeOH, 2oz. bulk jar	H <sub>2</sub> O & MeOH	<-10°C	48 hours to freeze, 14 days to analyze
Encore Kit: 3 x 5g Encore samplers, 4oz. bulk jar	NaHSO <sub>4</sub> , H <sub>2</sub> O, or MeOH (added in lab)	0-6°C	48 hours to preserve, 14 days to analyze
Encore Kit: 3 x 5g Encore samplers, 2 x pre-weighed 40-mL VOA w/ NaHSO <sub>4</sub> , pre-weighed 40-mL VOA w/ MeOH, 4oz. bulk jar	NaHSO <sub>4</sub> or MeOH	0-6°C	48 hours to preserve, 14 days to analyze

**Attachment 3:  
QC Summary**

QC Item	Frequency	Criteria	Corrective Action
Clock Time	EPA 8260B and EPA 8260C: 12 hours  EPA 624: 24 hours  EPA 624 Cluster Rule (Chloroform Only): 8 hours	Clock time starts with the injection of the BFB.  Analysis of samples and QC items must conclude within expiration of clock time. Subsequent analysis requires new BFB.	Not applicable
Tune Standard (BFB)	At beginning of each clock	Refer to Attachment 7.	- Perform instrument maintenance - Re-tune.
Trap Check Standard	At beginning of each clock	<RL (Chloromethane & Bromomethane)	- Perform instrument maintenance. - Change trap.

QC Item	Frequency	Criteria	Corrective Action
<p>Initial Calibration (ICAL)</p> <ul style="list-style-type: none"> <li>- Minimum of 3 points for EPA 624.</li> <li>- Minimum of 5 points for EPA 8260B and EPA 8260C.</li> </ul>	<p>Upon instrument set-up, and after unsuccessful CCV</p>	<p><u>EPA 624:</u> %RSD &lt; 35% If %RSD &gt; 35%, use curve fit w/ <math>r^2 &gt; 0.990</math>.</p> <p><u>EPA 8260B:</u> - CCC: %RSD &lt; 30% - SPCC: <math>RRF_{avg} &gt; \text{Attachment 5}</math> - If %RSD &gt; 15%, use curve fit with <math>r^2 &gt; 0.990</math>.</p> <p><u>EPA 8260C:</u> - 5-point Minimum RRF per Attachment 6. %RSD &lt; 20%. If %RSD &gt; 20%, use curve fit w/ <math>r^2 &gt; 0.990</math>; If linear fit, RL Level: 30% of true; If <math>r^2 &lt; 0.990</math>, use %RSD (allowed for &lt;10% of total # analytes; no analyte &gt;60% RSD).</p>	<ul style="list-style-type: none"> <li>-Reanalyze standard(s)</li> <li>-Prepare new standard(s) and reanalyze</li> <li>-Perform injector port maintenance and reanalyze standards</li> <li>-Retune and reanalyze standards</li> <li>-Replace column and reanalyze standards</li> <li>-Clean source and reanalyze standards</li> </ul>
<p>Initial Calibration Verification (ICV)</p> <ul style="list-style-type: none"> <li>- Second Source</li> </ul>	<p>After each ICAL</p>	<p><u>EPA 624:</u> Attachment 12</p> <p><u>EPA 8260B:</u> %RSD &lt; 30%; Poor Performer %D &lt; 60% as per Attachment 11</p> <p><u>EPA 8260C:</u> %D &lt; 30% Poor Performer %D &lt; 50% as per Attachment 11.</p>	<ul style="list-style-type: none"> <li>-Reanalyze standard</li> <li>-Prepare new standard and reanalyze</li> <li>-Recalibrate</li> </ul>

QC Item	Frequency	Criteria	Corrective Action
Continuing Calibration Verification (CCV)	After BFB	<u>EPA 624:</u> Attachment 12  <u>EPA 8260B:</u> - CCC: %D < 20% - SPCC: RRF > Attachment 5 - Non-CCC and non-SPCC: %D <20%; Poor Performers <60% as per Attachment 11  <u>EPA 8260C:</u> Meet minimum RF criteria in Attachment 6. %D < 20% Poor Performer %D <50% as per Attachment 11. An NCM is required to denote analytes outside criteria.	- Reanalyze standard - Prepare new standard and reanalyze - Recalibrate
Internal Standards (ISTD)	Spiked in all CCVIS, samples, and batch QC items	CCVIS: - Area -50% to +100% CCV in ICAL. - RT +/-10 seconds from ICAL.  Samples & batch QC items: - Area within -50% to +100% of previous CCVIS.	- Evaluate chromatogram, spectra, and integrations - Reanalyze extract - Perform instrument maintenance and reanalyze extract - Re-extract and reanalyze if sufficient sample available
Surrogate Compounds	Spiked in all samples and batch QC items.	Within MLG limits	- Evaluate chromatogram, spectra, and integrations - Reanalyze sample, if sufficient sample available
Analytical Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable

QC Item	Frequency	Criteria	Corrective Action
Method Blank (MB)	One per batch	<1/2RL	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient sample is provided for MS/MSD	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	EPA 8260B and EPA 8260C: One per batch  EPA 624: 1 per 10% of samples	Within TALS MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	Within TALS MLG Limits	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006  Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006

QC Item	Frequency	Criteria	Corrective Action
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007



## Attachment 4: Preventative Maintenance and Troubleshooting

### Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

### Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

### Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
Injector Port							X	Replace septum, sleeve, inlet seal, and washer (Recommend every 2 weeks)
Sparge Tubes							X	Clean (Recommend every 3 months)
Column							X	Change column (Recommend annually)

D=daily; W=Weekly; M=monthly; Q=Quarterly; SA=semi-annually; A=annually; AN=as needed

### Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

### Contingency Plan

Close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

**Attachment 5:  
EPA 8260B Calibration Criteria: SPCCs and CCCs****Calibration Check Compounds (CCCs)**

Initial Calibration	Continuing Calibration
<=30% RSD	<=20% difference from initial calibration

**System Performance Check Compounds (SPCCs)**

SPCC	Minimum RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	>0.10
1,1,2,2-Tetrachloroethane	0.30

Note: The CCC and SPCC criteria must be met even if the calibration curve option is used for quantitation. If the CCC and SPCC criteria do not pass, a new calibration curve must be prepared and analyzed.

**Calibration Check Compounds (CCCs)**

Vinyl chloride  
1,1-Dichloroethene  
Chloroform  
1,2-Dichloropropane  
Toluene  
Ethylbenzene

**System Performance Check Compounds (SPCCs)**

Chloromethane  
1,1-Dichloroethane  
Chlorobenzene  
Bromoform  
1,1,2,2-Tetrachloroethane

**Attachment 6:**  
**EPA 8260C Minimum Response Factors**

<b>Compound</b>	<b>Minimum RRF</b>
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
Freon-113	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
Cis-1,2-Dichloroethene	0.100
MTBE	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethene	0.100
Cyclohexanone	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
Cis-1,3-Dichloropropene	0.200
Trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
m,p-Xylene	0.100
o-xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300

1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

**Attachment 7:  
BFB Tune Criteria**

**EPA 8260B and EPA 8260C**

m/e	Abundance Criteria
50	8.0-40.0% of mass 95
75	30.0-66.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	50-120%% of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0% of mass 176

\*8260 criteria taken from CLP OLMO4.0 (January 1998)

**EPA 624**

m/e	Abundance Criteria
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	Base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	<2.0% of mass 174
174	>50% of mass 95
175	5.0-9.0% of mass 174
176	95.0-101.0% of mass 174
177	5.0-9.0% of mass 176

**Attachment 8:**  
**Target Compound Information: Ions and ISTDs**

Analyte	Quant Ion	Secondary Ions		ISTD
1,1,1,2-Tetrachloroethane	131	133	119	3
1,1,1-Trichloroethane	97	99	61	2
1,1,2,2-Tetrachloroethane	83	85	168	3
1,1,2-Trichloro-1,2,2-trifluoroethane	101	151	103	1
1,1,2-Trichloroethane	83	97	99	2
1,1-Dichloroethane	63	65	83	1
1,1-Dichloroethene	96	61	98	1
1,1-Dichloropropene	75	110	77	2
1,2,3-Trichlorobenzene	180	182		3
1,2,3-Trichloropropane	110	112		3
1,2,4-Trichlorobenzene	180	182	145	3
1,2,3-Trimethylbenzene	105	120	77	3
1,2,4-Trimethylbenzene	105	120	77	3
1,2-Dibromo-3-Chloropropane	157	75	155	3
1,2-Dichlorobenzene	146	148	111	3
1,2-Dichloroethane	62	49	64	2
1,2-Dichloroethene, Total	Sum of cis and trans isomers			
1,2-Dichloropropane	63	76	65	2
1,3,5-Trimethylbenzene	105	120	77	3
1,3,5-Trichlorobenzene	180	182	184	3
1,3-Dichlorobenzene	146	148	111	3
1,3-Dichloropropane	76	78	41	2
1,3-Dichloropropene, Total	Sum of cis and trans isomers			
1,4-Dichlorobenzene	146	148	111	3
1-Chlorohexane	41	43		3
1-Methylnaphthalene	142	141		3
2,2-Dichloropropane	77	41		1
2-Butanone (MEK)	43	72		1
2-Chloro-1,3-butadiene	53	88		1
2-Chloroethyl vinyl ether	63	65	106	2
2-Chlorotoluene	126	91	63	3
2-Hexanone	43	58		3
2-Methylnaphthalene	142	141		3
2-Nitropropane	43	41	39	2
3-Chloro-1-propene	76	41		1
3-Methylhexane	43	71		2
4-Chlorotoluene	126	91	63	3
4-Isopropyltoluene	119	134	91	3
4-Methyl-2-pentanone (MIBK)	43	57	58	2
Acetone	58	43		1
Acetonitrile	41	40		1
Acrolein	56	55		1
Acrylonitrile	53	52	51	1
Amyl acetate	70	61		3
Benzene	78	50		2



Analyte	Quant Ion	Secondary Ions		ISTD
Benzyl chloride	91	63		3
Bromobenzene	156	77	158	3
Bromoform	173	171	175	3
Bromomethane	96	94	79	1
Butadiene	54	39		1
Carbon disulfide	76	78		1
Carbon tetrachloride	117	119	121	2
Chlorobenzene	112	77	51	3
Chlorobromomethane	49	128	130	1
Chlorodibromomethane	129	127	131	3
Chloroethane	64	66		1
Chloroform	83	85	47	1
Chloromethane	50	52		1
Chloroprene	53	88		1
cis-1,2-Dichloroethene	96	61	98	1
cis-1,3-Dichloropropene	75	77	110	2
Cyclohexane	56	69	84	2
Cyclohexanone	55	42	98	3
Dibromomethane	93	174	95	2
Dichlorobromomethane	83	85	129	2
Dichlorodifluoromethane	85	87	101	1
Dichlorofluoromethane	67	69		1
Ethyl acetate	43	61	70	1
Ethyl acrylate	55	73		1
Ethanol	45	45		1
Ethyl ether (diethyl ether)	59	74	45	1
Ethyl methacrylate	69	41	39	3
Ethylbenzene	91	106	51	3
Ethylene Dibromide	107	109		2
Ethylene oxide	43	44		1
Furan	68	39		1
Hexachlorobutadiene	225	223	190	3
Hexane	57	41	43	1
Iodomethane	142	127		1
Isobutyl alcohol	43	41		2
Isopropyl acetate	61	59	87	1
Isopropyl ether	45	43		1
Isopropylbenzene	105	120	77	3
m,p-Xylene	106	91	77	3
Methacrylonitrile	67	52		1
Methyl acetate	43	74		1
Methyl acrylate	55	85		1
Methyl methacrylate	69	41		2
Methyl styrene	118	103	91	3
Methyl tert-butyl ether	73	57		1
Methylcyclohexane	83	55	98	2
Methylene Chloride	84	49	86	1
Naphthalene	128	102	51	3

Analyte	Quant Ion	Secondary Ions		ISTD
n-Butanol	56	41	43	1
n-Butyl acetate	43	56		2
n-Butyl acrylate	55	56		2
n-Butylbenzene	91	92	134	3
n-Heptane	43	57	71	1
N-Propylbenzene	120	91	65	3
o-Xylene	106	91	77	3
Pentachloroethane	167	130		3
Propene oxide	58	43		1
Propionitrile	54	55		1
sec-Butylbenzene	105	134	91	3
Styrene	104	78	103	3
Tert-amyl methyl ether	73	55	87	1
Tert-butyl alcohol	59	41		1
tert-Butylbenzene	119	91	134	3
Tert-butyl ethyl ether	59	87		1
Tetrachloroethene	164	166	168	3
Tetrahydrofuran	42	71		1
Toluene	92	91	65	2
trans-1,2-Dichloroethene	96	61	98	1
trans-1,3-Dichloropropene	75	77	110	2
trans-1,4-Dichloro-2-butene	53	88	89	3
Trichloroethene	130	95	132	2
Trichlorofluoromethane	101	103	105	1
Trichlorotrifluoroethane (Freon 113)	101	151	103	1
Trihalomethanes, Total	Sum of chloroform, dichlorobromomethane, dibromochloromethane, and bromoform			
Vinyl acetate	43	86		1
Vinyl chloride	62	64		1
Xylenes, Total	Sum of m/p- and o- isomers			

Analyte	Quant Ion	Secondary Ions		ISTD
<b>Surrogates</b>				
Dibromofluoromethane	113	81	111	1
Toluene-d8	98	100	70	2
p-Bromofluorobenzene	95	174	176	3
1,2-Dichloroethane-d4	65	67	102	1
<b>Internal Standards</b>				
Fluorobenzene	96	77		1
Chlorobenzene-d5	82	117	119	2
1,4-Dichlorobenzene-d4	152	115	150	3

Note: For a complete list of target analytes for each method, refer to the TALS Method Limit Groups (MLGs).

**Attachment 9:**  
**Instrument Configurations**

System	GC	MS	P/T	Autosampler	Trap	GC/MS Data System	Methods
MSA	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSB	Agilent 6890	Agilent 5973	Tekmar 3000	Tekmar Aquatek 70	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSC	Agilent 7890	Agilent 5975	EST Encon Evolution	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSL	Agilent 5890	Agilent 5972	Tekmar LSC 2000	Varian Archon	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B SO
MSM	Agilent 5890	Agilent 5972	Tekmar LSC 2000	Varian Archon	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B SO
MSO	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSP	Agilent 6890	Agilent 5973	EST Encon (dual)	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSAC	Agilent 6890	Agilent 5973	Tekmar 3000	Tekmar Aquatek 70	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B AQ EPA 8260C AQ

SOP No. SA-VO-004, Rev. 2

Effective Date: 12/01/14

Page No.: 48 of 60

<b>System</b>	<b>GC</b>	<b>MS</b>	<b>P/T</b>	<b>Autosampler</b>	<b>Trap</b>	<b>GC/MS Data System</b>	<b>Methods</b>
MSAA	Agilent 6890	Agilent 5973	EST Encon	EST 8100 Archon	Supelco VOCARB 3000	Agilent Chemstation	EPA 624 EPA 8260B AQ EPA 8260C AQ
MSAD	Agilent 6890	Agilent 5973	EST Encon	EST Centurion	Supelco VOCARB 3000	Agilent Chemstation	EPA 8260B AQ EPA 8260C AQ

## Attachment 10

### Standard Information and Recipes

Store standards in glass vials fitted with Teflon-lined caps in standards-only refrigerator at <-10C. Choose a container volume that will have minimal headspace when standard is added. Mark the level on the outside of the container so that evaporation of the solvent will be apparent.

The expiration dates represent the maximum time a standard can be maintained. Standards that exhibit obvious signs of degradation must be replaced sooner.

#### Routine Target Compounds

##### Stock Standard Mixes

Stock/Mix	TALS ID	Vendor/Part Number	Concentration (ug/mL)
8260 List 1/ Std #1 Mega Mix	567641	Restek 567641	2000 (majority of compounds)
8260 List 1/ Std #2 Ketones	567642	Restek 567642	10,000
8260 List 1/Std #3 Gases	567645	Restek 567645	2000
8260 List 1/ Std #4 2-CEVE	567643	Restek 567643	2000
8260 List 1/ Std #5 Acrolein	567644	Restek 567644	5000
8260 List 1/Std #6 Vinyl Acetate	567646	Restek 567646	4000
8260 IS/Surrogate Mix	567651	Restek 567651	150-250

Expiration:

Un-opened ampuls: manufacturer's expiration date

Opened ampuls: 1 month (Note: These ampuls are used to prepare the Working Standards and are typically consumed/disposed open opening.)

##### 8260 Working ISTD (TALS ID: 8260 ISTD)

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 IS/Surrogate Mix	5000	25	50

Solvent: P/T Methanol

Expiration: 1 Month

**8260 List 1  
(Mega Mix Working Standard)  
(TALS ID: 8260 MMix)**

Stock/Mix	Initial Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 1/ Std #1 Mega Mix	625	25	50
8260 List 1/ Std #2 Ketones	625		250
8260 List 1/Std #3 Gases	625		50
8260 List 1/ Std #4 2-CEVE	625		50
8260 List 1/Std #6 Vinyl Acetate	625		100

Solvent: P/T Methanol

Expiration: 2 weeks from date prepared

(Note: The 25mL final volume is split between five 5mL Mini-nert vials. Due to the volatility of the gas analytes, each vial is assigned a 1-week expiration date from the date that vial was opened, not to exceed 14 days from the preparation date.)

**Calibration Standards**

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
8260 MMix	2	10	20	40	100	200	400
8260 List 1/ Std #5 Acrolein	1	5	10	20	50	100	200
8260 ISTD	100	100	100	100	100	100	100
Volume of water (mL)	100	100	100	100	100	100	100
Target Compounds (ng)	5	25	50	100	250	500	1000
Internal Standards (ng)	250	250	250	250	250	250	250

Expiration: 24 hours



**8260 List 2 (Non-routine)**

**Stock Standard Mixes**

Stock/Mix	TALS ID	Vendor/Part Number	Concentration (ug/mL)
8260 List 2/Std #1 Additions	567647	Restek 567647	2000-50,000
8260 List 2/ Std #2	567719	Restek 567719	2000
8260 List 3/ Std #3 Cyclohexanone	567648	Restek 567648	20,000
SAV Custom Adds	CS-16915	Restek CS-16915	2000

**8260 Working ISTD  
(TALS ID: 8260 ISTD)**

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 IS/Surrogate Mix	5000	25	50

Solvent: P/T Methanol  
Expiration: 1 month

**8260 List 2  
(TALS ID: 8260L2)**

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
8260 List 2/Std #1 Additions	625	25	50-2500
8260 List 2/ Std #2	625		50
8260 List 3/ Std #3 Cyclohexanone	625		500

Solvent: P/T Methanol  
Expiration: 1 month

**8260 SAV Adds  
(TALS ID: 16915)**

Stock/Mix	Aliquot Volume (uL)	Final Volume (mL)	Final Concentration (ug/mL)
SAV Custom Adds	625	25	50

Solvent: P/T Methanol  
Expiration: 1 month

### Calibration Standards

Stock/Mix	1	2	3	4	5	6	7
	Aliquot (uL) to prepare CAL standard						
8260 List 2/Std #1 Additions	1	5	10	20	50	75	100
8260 List 2/ Std #2	1	5	10	20	50	75	100
8260 List 3/ Std #3 Cyclohexanone	1	5	10	20	50	75	100
8260 Sav Adds	1	5	10	20	50	75	100
8260 ISTD	50	50	50	50	50	50	50
Volume of water (mL)	50	50	50	50	50	50	50
Target Compounds (ng) (majority of compounds)	5	25	50	100	250	500	1000
Internal Standards (ng)	250	250	250	250	250	250	250

Note: Record calibration standard preparation in A/D batch.

Expiration: 24 hours

## **Attachment 11**

### **Poor Performers**

As indicated in EPA 8260B and/or via assessment of laboratory control sample recoveries and control charts, the compounds listed below are poor performers and/or behave erratically. These compounds will not be included in the LCS/LCSD/MS/MSD marginal exceedance count, provided their %R is >10%.

Additionally, these analytes have exceptions to the routine ICV and CCV requirements as outlined in the applicable section of the SOP.

Note: An NCM must be initiated to denote this situation.

#### **Poor Performer List**

1,1,2-Trichloro-1,2,2-Trifluoroethane	n-Butanol
1,2-Dibromo-3-Chloropropane	Naphthalene
1,2,3-Trichlorobenzene	Pentachloroethane
1,4-Dioxane	Propionitrile
2-Butanone	Trans-1,4-Dichloro2-butene
2-Chloroethyl vinyl ether	Trichlorotrifluoromethane
2-Hexanone	Vinyl Acetate
2-Methyl-2-Propanol (t-Butyl Alcohol)	
4-Methyl-2-Pentanone	
2-Nitropropane	
2-Propanol	
Acetone	
Acetonitrile	
Acrolein	
Acrylonitrile	
Allyl Chloride	
Carbon Disulfide	
Bromoform	
Bromomethane	
Chloroethane	
Chloromethane	
Cis-1,4-Dichloro2-butene	
Cyclohexanone	
Dichlorodifluoromethane	
Dichlorofluoromethane	
Ethanol	
Ethyl Acetate	
Ethyl Methacrylate	
Iodomethane	
Isobutyl Alcohol	
Isopropyl Alcohol	
Methacrylonitrile	
Methyl Acetate	
Methyl Methacrylate	
Methyl Cyclohexane	

**Attachment 12**  
**EPA 624 CCV and ICV Criteria**  
**(Q Table)**

Analyte	Criteria* (ug/L)
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Bromomethane	2.8-37.2
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-chloroethyl vinyl ether	D-44.8*
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,2-Dichlorobenzene	12.6-27.4
1,3-Dichlorobenzene	14.6-25.4
1,4-Dichlorobenzene	12.6-27.4
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
trans-1,2-Dichloroethene	13.9-26.1
1,1-Dichloroethene	10.1-29.9
1,2-Dichloropropane	6.8-33.2
cis-1,3-Dichloropropene	4.8-35.2
trans-1,3-Dichloropropene	10.0-30.0
Ethylbenzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9
Tetrachloroethene	14.7-25.3
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethene	13.3-26.7
Trichlorotrifluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

\*These values are given in ug/L (i.e., concentration ranges). The laboratory has converted these values into percent recovery limits to be used to assess CCV, ICV, LCS/LCSD, and MS/MSD.

**Attachment 13**  
**Glassware Cleaning Procedures**

**GLASSWARE CLEANING PROCEDURES**

**VOLATILES DEPARTMENT**

1. Rinse glassware 3 times thoroughly with DI water.
2. Place glassware, top-down, within storage rack and allow to air dry.
3. If glassware was used to prepare waste sample, use FL-70 and water to scrub glassware and follow previous steps.

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## Attachment 14 Data Entry Procedures



Work Instruction  
Page 1 of 4

### Data Entry Procedures for Soil Preparations and Dilutions (EPA 8260B, EPA 8260C, and EPA 8015C\_GRO)

#### Summary

For EPA 8260B, EPA 8260C, and EPA 815C\_GRO, the majority of soil samples are collected in 3 pre-weighed, pre-preserved vials. Two vials contain DI water or sodium bisulfate to be used for low-level analyses (i.e., analyzed straight). One vial contains methanol to be used for medium-level analyses (i.e., analyzed at dilution factor of 20 to 40, or higher). A preparation batch (i.e., a weight batch) is utilized to capture initial/final weights and volumes. Dilutions are captured via CHROM and are automatically transferred to the TALS analytical batch.

Note: The initial and final weight/volume entered in the TALS analytical batch must always be 5g and 5mL. The dilution factor is used to adjust calculations for Medium-Level soil samples. Adjustment/entry of initial volumes in TALS to reflect the MeOH sample aliquot (e.g., 125uL for a DF40) must not be performed as this will result in double-calculation of the dilution factor and miscalculation of final results.

#### Entry of Values in to the Weight Batch

1. Record the tare weight, which is found on the vial label, in the "Tare Weight" column.
2. Weigh the vial to determine the combined weight of the vial and sample. Enter into "Vial & Sample" column.
3. TALS automatically calculates Initial Amount by subtracting the Tare Weight from the Vial & Sample weight.
4. Enter purge volume in the "Final Amount" column. Note: Purge volume is always equivalent to 5mL.
5. Record preservative type in Worksheet Notes section - i.e., DI Water (DI), sodium bisulfate (SB), or Methanol (MEOH).

Sample	Preservative	Tare Weight	Vial & Sample	Initial Amount	Final Amount
1	DI	4.07755	34.60	7.046	5
2	SB	3.147	35.71	5.78	5
3	SB	3.151	35.06	7.05	5
4	SB	4.077285	35.48	6.154	5
5	SB	3.140	41.12	8.77	5
6	SB	3.140	40.76	8.76	5
7	MEOH	4.077749	34.23	5.081	5
8	SB	3.142	27.42	5.89	5
9	SB	3.122	40.27	8.78	5



**Work Instruction**  
Page 2 of 4

**Low-Level Soil Procedure**

Note: Vials designated as DI or SB are used for the Low-Level analysis.

In the CHROM Worklist, enter 5g for the Initial Wt./Vol. and 5g as the Final Wt./Vol. (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.

The screenshot shows the 'Open Batch' window in the CHROM Worklist software. The window has a menu bar (File, Edit, Display, Tools) and a toolbar. The main area is divided into several sections:

- Open Batch:** A list of samples is shown on the left, including sample numbers and names.
- Batch Information:** Fields for 'Batch Name', 'Batch Number', 'Batch Date', and 'Batch Time' are visible.
- Initial Wt./Vol. and Final Wt./Vol.:** These fields are both set to '5g' and are circled in red.
- Batch Settings:** Fields for 'Batch Size', 'Batch Type', 'Batch Unit', and 'Batch Weight' are visible.
- Batch Results:** Fields for 'Batch Results', 'Batch Results Unit', and 'Batch Results Weight' are visible.
- Batch Summary:** A summary of the batch information is shown on the right.





**Work Instruction**  
Page 3 of 4

**Medium-Level Soil Procedure**

Note: Vials designated as MEOH are used for the Medium-Level analysis. An aliquot of sample is taken from the MeOH vial and diluted in water.

The default (i.e., lowest) dilution for Medium-Level analyses is prepared as follows:

Method	Sample Aliquot (uL)	Final Volume (mL)	Dilution Factor
EPA 8260B / EPA 8260C	125	5	40
EPA 8015C GRO	250	10	20

Higher dilutions are prepared as follows:

Method	Sample Aliquot (uL)	Final Volume (mL)	Dilution Factor
EPA 8260B / EPA 8260C	100	5	50
EPA 8260B / EPA 8260C	50	5	100
EPA 8260B / EPA 8260C	25	5	200
EPA 8260B / EPA 8260C	10	5	500
EPA 8260B / EPA 8260C	5	5	1000
EPA 8015C GRO	100	10	50*
EPA 8015C GRO	50	10	100*
EPA 8015C GRO	20	10	250*
EPA 8015C GRO	10	10	500*
EPA 8015C GRO	5	10	1000*
EPA 8015C GRO	1	10	5000*
EPA 8015C GRO	0.5	10	10000*

\* = Due to differences in the autosampler types utilized for EPA 8260B and EPA 8260C versus EPA 8015C GRO, there is a difference in the water volume added to the sample to prepare the dilution. The autosamplers utilized for EPA 8260B and EPA 8260C add 5mL of water upon sampling of the vial. The autosamplers utilized for EPA 8015C GRO do not perform this action. As such, an additional 5mL of water is added to the EPA 8015C GRO sample vial upon preparation of the dilution (i.e., the dilution is prepared in 10mL of water).

Note: The dilution factor obtained is based on the 5mL purge volume. (e.g., for EPA 8015C GRO, a 250uL MeOH sample aliquot diluted into 10mL of water is equivalent to a dilution factor of 20.)

Note: Dilutions larger than those listed in the table require the use of a serial dilution.



**Work Instruction**  
Page 4 of 4

In the CHROM Worklist, enter 5g for the Initial Wt/Vol. and 5g as the Final Wt/Vol. (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.

The screenshot shows the CHROM Worklist software interface. The 'Initial Wt/Vol' and 'Final Wt/Vol' fields are highlighted with a red circle, showing values of 5g and 5g respectively. The interface includes a list of samples on the left, a central data entry area, and a right-hand panel with various settings and options.

Enter the dilution factor in CHROM to reflect the dilution prepared (as shown below). This information is automatically transferred to the worksheet section of the analytical batch in TALS.

In this example, a 5uL aliquot was taken from the MeOH vial and added to 5mL of water. For EPA 8260B analysis, this results in a dilution factor of 1000.

The screenshot shows the CHROM Worklist software interface. The 'Dilution Factor' field is highlighted with a red circle, showing a value of 1000. The interface includes a list of samples on the left, a central data entry area, and a right-hand panel with various settings and options.

## 18.0 **Revision History**

### Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made.
- Replaced references to LIMS with TALS.
- Add requirement to scan/attach standard COAs to TALS. Section 7.3
- Adjusted sample collection and storage conditions to reflect 0-6°C. Sections 8 and 16.1.1 and Attachment 2
- Removed requirement to recalibrate if trap check standard does not meet acceptance criteria. Section 9.2.2.3 and Attachment 3
- Removed requirement to perform IDOC from second source. Removed requirement to document IDOC using IDOC Cover Page. Section 12.5
- Included glassware cleaning procedures. Attachment 13
- Removed reference to maintenance contracts, as these are no longer maintained by the laboratory. Attachment 4
- Added additional instruments to Basic Instrument Configuration list. Attachment 9
- Removed allowance to use the Grand Mean Exception when evaluating ICAIs, ICVs, and CCVs. Adjusted text accordingly to better define new criteria. Section 9 and Attachment 3
- Updated Poor Performer list to be consistent with DOD QSM V5 Variance document. Attachment 11
- Clarified section on routine batch QC to denote that additional containers are seldom received for MS/MSD; therefore the laboratory routinely defaults to performing LCS/LCSD.
- Updated Target Compound Identification to include recent additions to analyte list. Attachment 8
- Added Data Entry Procedure Work Instruction. Attachment 14
- Added reference to screening process outlined in SOP SA-VO-001. Section 11.1.2

**CARBON CONTENT IN WATER:  
Total Carbon (TC), Total Organic Carbon (TOC), and Total Inorganic  
Carbon (TIC)**

**(Methods: EPA 9060, EPA 9060A, SM5310B, & EPA 415.1)**

**Approvals (Signature/Date):**

Andrea Teal September 06, 2013  
Date  
Quality Assurance Manager / Interim Environmental Health & Safety Coordinator

Carol Webb September 09, 2013  
Date  
Operations Manager – Inorganics

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**Facility Distribution No. 1**

**Distributed To: QA Navigator**

## 1.0 **Scope and Application**

This SOP gives the procedures for the determination of total carbon (TC), total inorganic carbon (TIC), and total organic carbon (TOC) in water samples by combustion.

Total carbon is comprised of total organic carbon and total inorganic carbon.

- If TOC is requested, the sample is acidified upon collection and analyzed.
- If TC is requested, an unacidified sample is collected and analyzed.
- If TIC is requested, the sample is collected and analyzed twice (once for the acidified portion and once for the unacidified portion) and the TOC result is subtracted from the TC result to obtain TIC.

Note: The determination of TOC involves purging the sample which eliminates any volatile organic compounds (VOCs) from the sample matrix. For this reason, the TOC reported is actually the non-purgeable TOC. The determination of TC or TIC involves analyzing the sample without purging the VOCs from the sample matrix.

This procedure can also be used for the determination of dissolved organic carbon (DOC), dissolved inorganic carbon (DIC), and dissolved carbon (DC) by performing a filtration on the sample prior to acidification and analysis.

The reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

## 2.0 **Summary of Method**

Total organic carbon (TOC) is measured by purging an acid-preserved sample to eliminate the inorganic carbon present in the sample. The acid converts any carbonates present in the sample to CO<sub>2</sub>, which is then purged out of the sample. The sample is then combusted at 680°C. The carbon is converted to carbon dioxide and is measured by an infrared (IR) detector.

Total carbon is performed in the same manner as TOC using unpreserved vials and eliminating the purging step. TIC is determined using a calculation by subtracting the TOC result from the TC result.

Dissolved organic carbon (DOC) is performed by filtering an aliquot of unpreserved sample with a 0.45µm filter and preserving the filtrate to a pH of <2 with acid. The filtered sample is analyzed using the same procedures and equipment as the TOC analysis.

The following equation shows the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

Where:

TC = total carbon

TOC = total organic carbon

TIC = total inorganic carbon

- 2.3 This SOP is based on the following methods: EPA Method 415.1, Standard Methods 5310B, EPA Method 9060, and EPA Method 9060A.

### 3.0 **Definitions**

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

### 4.0 **Interferences**

#### 4.1 **Procedural Interferences**

- 4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.
- 4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.
- 4.1.3 High purity reagents and solvents are used to help minimize interference problems. Sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.
- 4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

#### 4.2 **Matrix Interferences**

- 4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.
- 4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.
- 4.2.3 Sample particulates that are larger than the syringe will not be included in the carbon determination. This condition may cause low results if the particulates contain



measurable levels of carbon. The analyst must be cognizant of samples that may clog the sample syringe, and initiate an NCM, as applicable, to address this situation.

- 4.2.4 Liquid samples containing oil or that are oily in nature will deactivate the catalyst which may require regeneration or replacement prior to running other samples.
- 4.2.5 It is very important that the inorganic carbon be removed from the sample prior to the direct determination of the TOC (non-purgeable TOC). Failure to remove the inorganic component of the sample may result in artificially high TOC results.

## 5.0 **Safety**

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

### 5.1 **Specific Safety Concerns or Requirements**

- 5.1.1 The furnace on the carbon analyzer must be cooled to room temperature before maintenance is performed. The temperature of the analyzer furnace can reach 680°C. The analyst must be careful to avoid touching these very hot surfaces.

- 5.1.2 Care must be taken when handling sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and solutions of sulfuric acid.

Sulfuric acid is a strong oxidizer and is a corrosive. It will react violently when combined with organic compounds, possibly producing fire. Inhalation can cause irritation of the nose, throat, mucus membranes, and upper respiratory tract. Contact with the eyes can cause blurred vision, redness, pain, and even blindness.

- 5.1.3 The carrier gas MUST remain on while the instrument is in operation. The analyst must ensure that there is sufficient carrier gas available in the tank to complete the run. Failure to have carrier gas running through the instrument while the combustion furnace is at the operating temperature may result in melting of the combustion tubes.

### 5.2 **Primary Materials Used**



The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS.

Note: This list does not include all materials used in the procedure. A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS. Electronic copies of MSDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit <sup>1</sup>	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1mg/m <sup>3</sup> TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
<sup>1</sup> Exposure limit refers to the OSHA regulatory exposure limit.			
Note: Always add acid to water to prevent violent reactions.			

## 6.0 Equipment and Supplies

### 6.1 Equipment and Instrumentation

Shimadzu TOC-VCPN Carbon Analyzer

### 6.2 Analytical Data System / Software / Hardware

The TOC Control software is used on a Windows-based PC to schedule and acquire data and to output the data to the laboratory's LIMS system (i.e., TALS). This software allows for the construction of a calibration curve and calculation of concentrations of analytes.

### 6.3 Lab Supplies

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Disposable Graduated Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Support Equipment (Verification and Use)*

Disposable Transfer Pipettes – various sizes

Filters – 0.45um, disposable

Syringes – 10mL, disposable

Autosampler vials – 25mL

pH paper

Detergent – Liquinox, used for washing non-disposable labware.

Ultra pure compressed air, regulator, and appropriate fittings – The compressed air must contain less than 1ppm of carbon dioxide. This gas is used as the carrier and sparge gas for the analysis of aqueous samples.

Stir plate

Stir bars – Teflon

#### 6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The sample collection containers supplied by the laboratory are purchased with Certificate of Analysis attesting to purity. The routine bottle kits supplied are as follows:

Analyte	Bottle Type	Preservative	Other Equipment
TOC	40mL amber VOA	H <sub>2</sub> SO <sub>4</sub>	None
TC	40mL VOA	None	None
TIC	40mL amber VOA	H <sub>2</sub> SO <sub>4</sub>	None
	40mL VOA	None	None
DOC	40mL amber VOA	H <sub>2</sub> SO <sub>4</sub>	10mL syringe 0.45um filter
DC	40mL VOA	None	10mL syringe 0.45um filter
DIC	40mL amber VOA	H <sub>2</sub> SO <sub>4</sub>	10mL syringe 0.45um filter
	40mL VOA	None	10mL syringe 0.45um filter

## 7.0 Reagents and Standards

### 7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

## 7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*.

Sulfuric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

### 7.2.1 Laboratory Reagent Water – ASTM Type II

### 7.2.2 Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) – concentrated, reagent grade or better. Store in original container at room temperature.

Storage: reagent cabinet

Expiration: manufacturer's expiration date or 2 years from the date opened, whichever is sooner

### 7.2.3 Sulfuric acid solution (50% $\text{H}_2\text{SO}_4$ ) – Add approximately 400mL of laboratory reagent water to a 2L beaker. Place the beaker on a magnetic stir plate located under a hood. Add a Teflon stir bar to the laboratory reagent water and turn the stir plate on. Slowly add 500mL of concentrated $\text{H}_2\text{SO}_4$ to the beaker in small aliquots. After the entire 500mL of $\text{H}_2\text{SO}_4$ has been added, continue mixing and allow the solution to cool. After the solution has cooled, transfer to a 1L volumetric flask and dilute to volume with laboratory reagent water. This solution must be stored in a glass container at room temperature. **Caution: Sulfuric acid is extremely toxic, has a suffocating odor, and can cause severe chemical burns.**

Storage: Acid storage cabinet, away from incompatibles

Expiration: 2 years from the date prepared or the expiration date of the parent reagent, whichever is sooner

## 7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Traceability*. Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the Public\_QA Drive.

### 7.3.1 Potassium Hydrogen Phthalate (KHP) Salt – Primary source; reagent grade; purchased from EMD Chemicals

Storage: Store in original container at room temperature in a desiccator

Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

Note: This material must be lightly crushed, to break up lumps, and dried at 120°C prior to preparation of the stock standard.

- 7.3.2 ICAL/MS/MSD Stock Standard, 2000mg/L – Prepared by adding 4.256g of primary source KHP Salt to 1L reagent water.

Storage: Refrigerated at 4°C

Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

- 7.3.2 Calibration Standards

Prepare the calibration standards according to the recipe in the following table. Smaller volumes and different concentrations may be prepared if approved by the department manager.

Calibration Standard Level	ICAL/MS/MSD Stock Standard Volume (mL)	Final Volume (mL)	Calibration Standard Concentration (mg/L)
1	0.0	1000	0.0
2	0.50	1000	1.0
3	2.5	1000	5.0
4	5.0	1000	10
5	12.5	1000	25
6	25	1000	50
7	50	1000	100

Adjust the pH of each standard to less than 2 with 50% H<sub>2</sub>SO<sub>4</sub>. Transfer the calibration standards to amber storage bottles.

Storage: Refrigerated at 4°C

Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

- 7.3.3 Initial Calibration Verification (ICV)

Second Source Potassium Hydrogen Phthalate (KHP) Salt – Reagent grade, purchased from Fisher Scientific. Note: The ICV standard must be purchased from a source independent of that used to prepare the calibration standards. Store in original container at room temperature.

Storage: Desiccator

Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner

Note: This material must be lightly crushed, to break up lumps, and dried at 120°C prior to preparation of the stock standard.

- 7.3.4 ICV Stock Standard – Transfer 4.256g of the Second Source KHP to a 1L volumetric and dissolve in a small amount of laboratory reagent water. Dilute to volume with laboratory reagent water. The stock standard has a concentration of 2000mg C/L. Transfer the stock standard to an amber storage bottle. Storage: Refrigerated at 4°C

Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner

- 7.3.5 Laboratory Control Sample (LCS) Solution (20mg/L) – Add 10mL ICV Stock Standard to 1L reagent water. Adjust the pH to less than 2 with 50% H<sub>2</sub>SO<sub>4</sub>. Transfer the stock standard to an amber storage bottle.  
Storage: Refrigerated at 4°C  
Expiration: 3 months from the date prepared or the expiration date of the parent reagent, whichever is sooner
- 7.3.6 Sodium Carbonate (NaCO<sub>3</sub>) – reagent grade. Store in original container at room temperature.  
Storage: reagent cabinet  
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner
- 7.3.7 Sodium Bicarbonate (NaHCO<sub>3</sub>) – reagent grade. Store in original container at room temperature.  
Storage: reagent cabinet  
Expiration: manufacturer's expiration date or 5 years from the date opened, whichever is sooner
- 7.3.8 Carbonate/Bicarbonate TIC Check Solution – Place approximately 800mL of laboratory reagent water in a 1L volumetric flask. Add 1.38g NaHCO<sub>3</sub> and 1.77g NaCO<sub>3</sub> and mix until all solid is dissolved into solution. Dilute to 1L with laboratory reagent water and adjust the pH to <2 with 50% H<sub>2</sub>SO<sub>4</sub>. Transfer to an amber glass container and store at room temperature.  
Storage: reagent cabinet  
Expiration: 2 years from the date prepared or the expiration date of the parent reagent, whichever is sooner

## **8.0 Sample Collection, Preservation, Shipment, and Storage**

### **8.1 Aqueous Samples**

All aqueous samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of analysis. Samples must be analyzed within 28 days of collection.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives. NCMs must be initiated for samples for TC and/or TIC analysis that are received containing headspace.

#### **8.1.1 TOC**

Aqueous samples for TOC are routinely collected in 40mL amber VOA vials containing sulfuric acid preservative. This preservative should be sufficient to achieve a sample pH of <2.

#### **8.1.2 TC**

Aqueous samples for TC are routinely collected in 40mL VOA vials with no preservative.

The 40mL VOA must be filled with no headspace to minimize the loss of the volatile organic components.

#### 8.1.3 TIC

Aqueous samples for TIC require 2 sets of bottles to be supplied:

- 1 set as outlined above for TOC
- 1 set as outlined above for TC

#### 8.1.4 DOC

Aqueous samples for DOC should be filtered and preserved in the field, within 2 hours of collection. The samples are filtered using a 0.45um filter, and the filtrate should be collected in a 40mL amber VOA vial containing sulfuric acid preservative sufficient to achieve a sample pH of <2.

Note: Samples for DOC may be collected in unpreserved containers, filtered in the lab upon receipt, and then preserved with H<sub>2</sub>SO<sub>4</sub>; however, this is not recommended as the 2-hour holding time will not be met. An NCM must be initiated for samples collected and preserved in this manner.

#### 8.1.5 DC

Aqueous samples for DC should be filtered in the field, within 2 hours of collection, using a 0.45um filter. The filtrate should be collected in a 40mL VOA vial with no preservative. The 40mL VOA must be filled with no headspace to minimize the loss of the volatile organic components.

#### 8.1.6 DIC

Aqueous samples for DIC require 2 sets of bottles to be supplied:

- 1 set as outlined above for DOC
- 1 set as outlined above for DC

#### 8.1.7 Preservation Checks

##### 8.1.7.1 pH Verification

For each preserved sample,

- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is greater than 2, initiate a Nonconformance Memo. Adjust the sample pH to <2 using H<sub>2</sub>SO<sub>4</sub>.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

## 9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

### 9.1 Batch QC

An analytical batch consists of up to 20 environment samples and the associated quality control items. The minimum QC items required for each analytical batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or sample duplicate (SD).

Note: Method SM5310B recommends an LFB spiked at the MRL to be performed quarterly (preferably daily). Therefore, if performing this method, a low-level LCS (spiked at the RL) is required per batch.

If there is insufficient sample to perform the MS or sample duplicate, an NCM must be initiated and the LCS must be prepared in duplicate (i.e., LCSD must be performed).

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

Batch QC must meet the criteria given in Attachment 4 of this SOP.

### 9.2 Instrument QC

#### 9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards.



The initial calibration standard concentrations currently in use in the laboratory are as follows:

Calibration Standard Level	ICAL/MS/MSD Stock Standard Volume (mL)	Final Volume (mL)	Calibration Standard Concentration (mg/L)
1	0.0	1000	0.0
2	0.50	1000	1.0
3	2.5	1000	5.0
4	5.0	1000	10
5	12.5	1000	25
6	25	1000	50
7	50	1000	100

Refer to Section 7.3.2 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

The purge is turned off to analyze the initial calibration. Each initial calibration standard is analyzed by the instrument up to 5 times. The instrument chooses the best 3 of the 5 analyses and averages this result to determine the response for the associated concentration.

#### 9.2.1.1 ICAL Criteria

The correlation coefficient (r) of the regression curve must be greater than 0.995 for the initial calibration curve to be acceptable.

Note: The calibration must be performed quarterly (i.e., once every 3 months) at a minimum.

#### 9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-16 with a standard obtained from a second source.

The initial calibration verification standard concentration currently in use in the laboratory is 20mg/L. Refer to Section 7.3.3 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

The ICV must be within +/-10% of the true value to be acceptable.

#### 9.2.3 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks are analyzed at the beginning and end of each batch.

Initial and continuing calibration blanks must be  $<1/2RL$  to be acceptable.

#### 9.2.4 Continuing Calibration Verification

The initial calibration curve must be verified every 10 analyses with a mid-level standard.

The CCV must be within  $\pm 10\%$  of the true value to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to the 50mg/L level of the ICAL. Refer to Section 7.3.2 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

#### 9.2.5 Carbonate/Bicarbonate TIC Check Solution

This solution is analyzed once daily as a check of the efficiency of the acidification and sparging to remove inorganic carbon from samples being analyzed for TOC.

Transfer an aliquot of the Carbonate/Bicarbonate TIC Solution to a labeled autosampler vial.

Adjust the pH of the solution to  $<2$  with 50%  $H_2SO_4$  and analyze.

Evaluate the result. If the TIC Check standard is less than the RL, continue with the sequence. If not, check the pH of the standard to make sure that it is less than 2.0, remake the standard, and reanalyze the standard. If the result is still greater than 2.0, contact the supervisor immediately. The instrument may need to be serviced.

#### 9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 4. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

### 10.0 **Procedure**

#### 10.1 Sample Preparation

Remove the samples from the refrigerator and allow them to come to room temperature.

#### 10.2 QC Sample Preparation

Prepare the method blank by adding 25mL of laboratory reagent water that has been adjusted to a pH of <2 with 50% H<sub>2</sub>SO<sub>4</sub> to an autosampler vial.

Prepare the LCS by adding 25mL of LCS Solution to an autosampler vial.

Prepare the matrix spikes (MS and MSD) by adding 0.25mL of the 2000mg/L ICAL/MS/MSD Stock Standard Solution to 25mL of sample.

Prepare a sample duplicate.

### 10.3 Analysis

#### 10.3.1 Instrument Operating Conditions

The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 2 of this SOP.

Turn the Shimadzu TOC-VCPN on and set the following parameters:

Analyzer Furnace: 680°C  
Carrier Gas: 150mL/min (ultrapure compressed air)  
Purge Gas: 50mL/min (ultrapure compressed air)  
Minimum Number of Replicates: 3  
Maximum Number of Replicates: 5

Allow the instrument to equilibrate and the furnace to reach the set point before processing samples.

#### 10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

#### 10.3.3 Sample Analysis

The sample must be analyzed using the same volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

Each sample must be analyzed in quadruplicate for EPA 9060 and EPA 9060A, and in triplicate for SM5310B and EPA 415.1. For EPA 9060 and EPA 9060A, the instrument collects/excludes replicates until it obtains 4 replicates with 10% precision. For EPA 415.1 and SM5310B, the instrument collects/excludes replicates until it obtains 2 sequential replicates with 10% precision.

If a sample contains a concentration of TOC greater than the linear range of the calibration curve on range 3, the sample must be diluted and re-analyzed. The average of the quadruplicate analyses results is reported as the final result in LIMS.

Note: The laboratory purges the acidified sample during analysis to remove the inorganic and purgeable organic carbon from the sample matrix; therefore, the non-purgeable TOC (nTOC) or non-purgeable DOC (nDOC) concentration is determined.

In order to determine the total carbon (TC) or dissolved carbon (DC) concentration an aliquot of the un-preserved sample is analyzed without purging the inorganic and purgeable organic carbon from the sample.

#### 10.3.4 Example Analytical Sequence

An example analytical sequence is listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source: 50mg/L
ICB	Used as method blank
Bicarbonate/Carbonate TIC Check Standard	
LCS	
Samples	Up to 10 sample analyses, including MS/MSD
CCV	5mg/L
CCB	
Samples & Batch QC Items	Up to 10 analyses, including MS/MSD
CCV	50mg/L
CCB	
Samples & Batch QC Items	Up to 10 sample analyses, including MS/MSD
CCV	50mg/L
CCB	

## 11.0 Calculations / Data Reduction

### 11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

#### 11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

#### 11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total Results  $\geq$  Dissolved results
- COD  $\geq$  TOC
- TC  $\geq$  TOC + TIC

#### 11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The following equations show the relationship between the various forms of carbon that can be measured with this procedure:

$$TC = TOC + TIC$$

Where:

TC = total carbon

TOC = total organic carbon

TIC = total inorganic carbon

$$DC = DOC + DIC$$

Where:

DC = dissolved carbon

DOC = dissolved organic carbon

DIC = dissolved inorganic carbon

11.2.3 The calculation to determine final concentration is given as follows:

$$C(\text{mg} / \text{L}) = C_{\text{curve}} \otimes DF$$

Where:

$C_{\text{curve}}$  = concentration of TOC from curve

DF = dilution factor

## 12.0 **Method Performance**

### 12.1 **Reporting Limit Verification (RLV)**

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter.

### 12.2 **Method Detection Limit (MDL) Study**

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

### 12.3 **Method Detection Limit Verification (MDLV)**

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

12.4 Note: RLVs, MDL Studies, and MDLVs are performed for TOC and TC, only. The TOC and TC values are used to satisfy the DOC and DC values, respectively. TIC and DIC are obtained using a calculation; therefore, these values are defined as the higher of the values between TOC and TC (or DOC and DC).

### 12.5 **Demonstrations of Capability**

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful

completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The LCSs must be from a second source than that used to prepare the calibration standards. The IDOC must be documented on the IDOC Form shown in SOP SA-QA-006 with documentation routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), or via acceptable results on a PT study. The CDOC must be documented and routed to the QA Department for filing.

#### 12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

### 13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the TestAmerica Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

### 14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

#### 14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess samples, reagents, and standards must be disposed in accordance with the TestAmerica Savannah addendum to the Environmental Health and Safety Manual.
- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department



for disposal.

## 15.0 **References / Cross-References**

- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-AN-100: *Support Equipment (Verification and Use)*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006: *Training Procedures*
- SOP SA-QA-07: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Method for Chemical Analysis of Water and Wastes*; U.S. EPA Office of Research and Development: Cincinnati, Ohio, March, 1983
- *Test Methods for the Evaluating Solids Wastes, Third Edition, SW-846*; US EPA Office of Solid Waste and Emergency Response: Washington, DC. (including Updates III and IV)
- *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association; Washington, D.C.
  - SM5310B: Total Organic Carbon (Approved 2000; Editorial Revisions 2011)

## 16.0 **Method Modifications and/or Clarifications**

- 16.1 Standard Methods 5310B recommends using phosphoric acid to preserve samples and standards; the EPA methods recommend hydrochloric acid. The preservation technique performed by the laboratory incorporates sulfuric acid.
- 16.2 The reference methods do not specify a holding time to be used for TC and TIC. The laboratory defaults to 28 days for these analytes (and DC and DIC).
- 16.3 The laboratory allows for lab filtration of DOC samples; however, an NCM must be utilized to indicate the 2-hour holding time was not met.
- 16.4 MDLs are determined for TOC and TC, only. These TOC and TC MDLs are used to satisfy the DOC and DC MDLs, respectively. TIC and DIC are obtained using a calculation; therefore, these MDL values are defined as the higher of the values between TOC and TC (or DOC and DC).
- 16.5 SM5310B specifies that a blank, and an LCS or MS must be performed per 10 analyses with a 2<sup>nd</sup> source standard. The laboratory uses a same source standard for the MS; however, a second source LCS is utilized.
- 16.6 The SM5310B reference method recommends a LFB at the MRL to be performed quarterly (preferably daily). The laboratory meets these requirements by preparing an

LCS at the RL in each batch for SM5310B. The reference documents do not specify criteria for the low-level LCS; therefore, the laboratory defaults to 50-150%.

## **17.0 Attachments**

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

Attachment 1: Sample Collection, Preservation, and Holding Time Table

Attachment 2: Preventative Maintenance and Troubleshooting

Attachment 3: SOP Summary

Attachment 4: QC Summary

## Attachment 1: SOP Summary

### Sample Preparation and Analysis Summary

Total organic carbon (TOC) is measured by purging the acid-preserved sample to eliminate the inorganic carbon present in the sample. The acid converts any carbonates present in the sample to CO<sub>2</sub>, which is then purged out of the sample. Purging the sample also eliminates any volatile organic compounds (VOC) from the sample matrix; therefore, any direct measurement of TOC is actually non-purgeable TOC. The sample is then combusted at 680°C. The carbon is converted to carbon dioxide and is measured by an infrared (IR) detector. Standards are analyzed under the same instrument conditions as the samples.

Total carbon and total inorganic carbon are performed in the same manner as TOC using unpreserved vials and eliminating the purging step.

Dissolved organic carbon (DOC) is prepared either in the field or immediately upon receipt in the laboratory by filtering an aliquot of unpreserved sample with a 0.45um filter and preserving the filtrate to a pH of <2 with 50% H<sub>2</sub>SO<sub>4</sub>. The dissolved organic carbon is analyzed using the same procedures and equipment as the TOC analysis. The QC samples required for DOC are a filtered method blank and matrix spikes on a filtered sample.

### Analytical Sequence

An example analytical sequence is listed below.

Description	Comments
Blank	
Initial Calibration	
ICV	Second Source: 20mg/L
ICB	Used as method blank
Bicarbonate/Carbonate TIC Check Standard	
LCS	
Samples	Up to 10 sample analyses, including MS/MSD
CCV	5mg/L
CCB	
Samples & Batch QC Items	Up to 10 analyses, including MS/MSD
CCV	50mg/L
CCB	
Samples & Batch QC Items	Up to 10 sample analyses, including MS/MSD
CCV	50mg/L
CCB	

**Attachment 2:**  
**Sample Collection, Preservation, and Holding Time Table**

Analyte	Routine Bottle Type	Other Field Equipment	Routine Volume	Minimum Volume	Chemical Preservative	Dechlorination Agent	Thermal Preservative	Holding Time
TOC	40mL VOA Vial	None	25mL	25mL	H <sub>2</sub> SO <sub>4</sub>	None	4°C <sup>1</sup>	28 Days from Collection
TC	40mL VOA Vial	None	25mL	25mL	None	None	4°C <sup>1</sup>	28 Days from Collection
TIC	40mL VOA Vial	None	25mL	25mL	H <sub>2</sub> SO <sub>4</sub>	None	4°C <sup>1</sup>	28 Days from Collection
	40mL VOA Vial	None			None	None	4°C <sup>1</sup>	28 Days from Collection
DOC	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	H <sub>2</sub> SO <sub>4</sub>	None	4°C <sup>1</sup>	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
DC	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	None	None	4°C <sup>1</sup>	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
DIC	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	H <sub>2</sub> SO <sub>4</sub>	None	4°C <sup>1</sup>	Filter: 2 Hours from Collection Analysis: 28 Days from Collection
	40mL VOA Vial	10mL syringe 0.45um filter	25mL	25mL	None	None	4°C <sup>1</sup>	Filter: 2 Hours from Collection Analysis: 28 Days from Collection

<sup>1</sup>Samples must be collected on ice and maintained at 0-6°C until time of analysis.

**Attachment 3:  
QC Summary**

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)  - Minimum of 4 points	Upon instrument set-up, and after unsuccessful CCV  - Quarterly, at a minimum	$r^2 > 0.995$	Re-calibrate
Second Source Initial Calibration Verification (ICV)	After each ICAL	Within $\pm 10\%$ of the true value	Re-calibrate
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and every 10 samples	Within $\pm 10\%$ of the true value	Refer to SOP SA-QA-016
Initial and Continuing Calibration Blank (ICB & CCB)	After ICV and every CCV	$< 1/2RL$	Terminate the analysis; correct the problem; and reanalyze the previous 10 samples
Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 10 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	$< 1/2RL$	Refer to SOP SA-QA-017
Laboratory Control Sample (LCS)	One per batch	Within MLG Limits	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per extraction batch, when insufficient sample is provided for MS/MSD/SD	Within MLG Limits	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS)	One per batch	50-150%	Refer to SOP SA-QA-017
Matrix Spike (MS)	One per extraction batch	Within MLG Limits	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Matrix Spike Duplicate (MSD)	One per extraction batch	Within MLG Limits	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006  Note: Unsupervised work must not begin until successful IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-06	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-07	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-07	Refer to SOP SA-QA-007



#### Attachment 4: Preventative Maintenance and Troubleshooting

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning.

An extensive spare parts inventory is maintained for routine repairs, consisting of combustion tubes, halogen scrubber, sample needles, o-rings, and platinum catalyst., and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

#### Preventive Maintenance

LABORATORY EQUIPMENT PREVENTIVE MAINTENANCE SCHEDULE								
Equipment / Item	Service Interval							Service
	D	W	M	Q	SA	A	AN	
Combustion tube							X	Replace as needed
Carrier gas pressure gauge	X							Check daily and replace gas as necessary
Humidifier water level	X							Check daily and refill as necessary

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually;  
AN = as needed

#### Desiccator Maintenance

##### Upright Desiccators with Doors

The following checks must be performed daily:

- Desiccant is active
- Hygrometer is in the low humidity zone
- Door is making an air tight seal

The desiccator door must remain closed and seated whenever possible.

When the desiccant turns from blue to light purple, discard desiccant, in accordance with the TestAmerica Savannah Addendum to the EHSM, and refill pan with fresh desiccant.

#### Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

### **Contingency Plan**

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

### **Maintenance Log**

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

### **Instrument Labeling**

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

## 18.0 **Revision History**

Summary of Changes from Previous Revision:

- Added requirement to perform LFB at MRL for method SM5310B. This change stems from SC DHEC Technical Deficiency. Section 9.1, Section 16.6, and Attachment 3.
- Removed references to HCl and replaced with H<sub>2</sub>SO<sub>4</sub>. This reagent is now used to adjust pH in standards and samples. Revised Method Modification text in section 16.1.
- Revised method reference to reflect 2011 revision of the methods in the 22<sup>nd</sup> Edition of Standards Methods. This change stems from a SC DHEC Technical Review Deficiency. The following method reference was revised:  
*Standard Methods for the Examination of Water and Wastewater*, American Public Health Association; Washington, D.C.
  - SM5310B: Total Organic Carbon (Approved 2000; Editorial Revisions 2011)

## LIQUID PREPARATION PROCEDURES FOR ICP AND ICP/MS

(Methods: EPA 200.7, 200.8, 3005A, 3010A, SM3030C, and Filtration)

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## 1.0 **Scope and Application**

This SOP gives the procedures for the preparation of metals in water and leachate samples prior to analysis by ICP (SOP SA-ME-070: *Elements by ICP*) or ICP/MS (SOP SA-ME-074: *Elements by ICP/MS*).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

## 2.0 **Summary of Method**

- 2.1 Total Recoverable Metals: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.
- 2.2 Total Metals and TCLP/SPLP Leachates: A known volume of sample, usually 50mL, is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA 3010A, and is utilized as the total metals preparation procedure for the EPA 200.7 and EPA 200.8.
- 2.3 Acid Extractable Metals: A known volume of sample, usually 50mL, preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. This procedure is equivalent to Standard Methods 3030C.
- 2.4 Silica Samples for EPA 200.7 and EPA 6010C: Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.
- 2.5 Drinking water samples for EPA 200.7 and EPA 200.8 with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA 200.7 or the EPA Method 200.8 preparation procedure is used.
- 2.6 Samples filtered for the determination of dissolved metals do not require digestion if the following criteria are met:
  - 1) The sample has a low COD (<20mg/L);
  - 2) The sample has a turbidity <1NTU ;
  - 3) The sample is colorless with no significant odor ; **and**
  - 4) The sample is of one liquid phase and is free of suspended particulates or precipitates after acidification.

Note: Generally the lab will digest samples for dissolved metals, even if the above criteria are met, as the presence of organics in the native sample may lead to false positives for arsenic and selenium.

- 2.7 This SOP is based on the following methods: EPA 200.7, EPA 200.8, EPA 3005A, EPA 3010A, and Standard Methods 3030C.

### **3.0 Definitions**

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

### **4.0 Interferences**

#### **4.1 Procedural Interferences**

- 4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.
- 4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.
- 4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.
- 4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

#### **4.2 Matrix Interferences**

- 4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require cleanup or dilution prior to analysis to reduce or eliminate the interferences.
- 4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.
- 4.2.3 Turbidity Matrix Interferences
- 4.2.3.1 The presence of floating debris and coarse sediments which settle out rapidly will give a low reading. Air bubbles will affect the results positively.

- 4.2.3.2 The presence of true color, that is the color of water which is due to dissolved substances that absorb light, will cause turbidities to be low, although this effect is generally not significant with drinking waters.
- 4.2.3.3 Light absorbing materials such as activated carbon in significant concentrations can cause low readings.

## 5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

### 5.1 Specific Safety Concerns or Requirements

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

### 5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

**NOTE: This list does not include all materials used in the procedure.** A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP.

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies



of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit <sup>1</sup>	Signs and Symptoms of Exposure
Hydrochloric Acid <sup>2</sup>	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid <sup>2</sup>	Corrosive Oxidizer Poison	2ppm TWA  4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
<sup>1</sup> Exposure limit refers to the OSHA regulatory exposure limit.			
<sup>2</sup> Always add acid to water to prevent violent reactions.			

## 6.0 Equipment and Supplies

### 6.1 Equipment and Instrumentation

Thermometers – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

Digestion block – capable of maintaining a sample digestion temperature of 95±5°C. The temperature of the digestion block must be monitored and recorded for each batch. The temperature is measured in a beaker or digestion vessel containing reagent water.

Turbidimeter – Hach Model 2100AN.

### 6.2 Volumetric Containers

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to Attachment 6 for Labware Cleaning Procedures.

Volumetric Labware	Volume	Type (Quantitative / Qualitative)	Use	Verification Frequency	Laboratory Verification Criteria
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Preparing Standards and Dilutions	None (Class A)	None (Class A)
Graduated Cylinders (Class A)	Various	QUANTITATIVE	Preparing Standards and Dilutions	None (Class A)	None (Class A)
Eppendorf-Style Mechanical Pipettes	Various	QUANTITATIVE	Preparing Standards and Dilutions	Monthly (Daily for DOD)	Accuracy = 2% Precision = 1%
Pump-Style Pipettes	Various	Qualitative	Acid/Base Delivery for pH Adjustment	Initially	Accuracy = 10% Precision = 10%
Autosampler Tubes	14mL	Qualitative	Digest Analysis	None	None
Digestion Vials	50mL	QUANTITATIVE	Initial and Final Volumes	None (Class A)	None (Class A)
Digestion Vials	100mL	QUANTITATIVE	Initial and Final Volumes	None (Class A)	None (Class A)
Disposable Medicine Cups	300mL	Qualitative	Used to contain aliquot for pH measurement	None	None

### 6.3 Lab Supplies

pH paper

Detergent – Alconox or equivalent, used for washing non-disposable labware.

0.45um polypropylene filters and syringes

### 6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are 250mL plastic containers purchased with Certificate of Analysis attesting to purity.

Note: Samples collected for the Lead and Copper Rule require 1 liter containers.

## 7.0 Reagents and Standards

### 7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard

or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it; that is, the "children may not outlive the parents".

## 7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*.

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

Laboratory Reagent Water – ASTM Type I

Nitric acid (HNO<sub>3</sub>): reagent grade. Stable under ordinary conditions of use and storage. Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Expiration:

Unopened: Manufacturer's expiration date

Opened: 5 years from date of opening

Hydrochloric acid (HCl): reagent grade. Stable under ordinary conditions of use and storage.

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.

Expiration:

Unopened: Manufacturer's expiration date

Opened: 5 years from date of opening

## 7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

### 7.3.1 Turbidity Standards

Refer to SOP SA-GE-206 for information on the turbidity standards.

### 7.3.2 Silica/Silicon Standards

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

- 7.3.2.1 Stock SiO<sub>2</sub> Standard, 10000mg/L Si / 21400mg/L SiO<sub>2</sub> – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's

expiration date.

Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.

- 7.3.2.2 Intermediate SiO<sub>2</sub> Standard, 467mg/L Si / 1000mg/L SiO<sub>2</sub> – Add 20mL to 30mL of reagent water to a clean, plastic 100-mL volumetric flask. Add the volume of the Stock SiO<sub>2</sub> Standard given in the table below to the volumetric flask. Dilute to volume with reagent water.

Element	Conc. Stock SiO <sub>2</sub> Std (mg/L)	Volume Stock SiO <sub>2</sub> Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> ) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

Storage: Store in a cool, dry, ventilated storage area. Store away from sunlight, heat, water, and incompatible materials.

Expiration: Prepare this solution every 180 days or sooner as needed or required. The expiration date must not exceed the expiration date of any of the components.

### 7.3.3 ICP/MS Spiking Solutions

- 7.3.3.1 ICP/MS Spiking Solution 1 – Purchased from CPI International (4400-060117RHO2). The concentrations of the analytes in this solution are listed on the accompanying certificate of analysis.

Storage: Store this solution at room temperature.

Expiration: Replace this solution by the manufacturer's expiration date or sooner if needed or required.

- 7.3.3.2 ICP/MS Spiking Solution 2 – Add 10mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Mercury (Hg)	1000	0.25	500	0.50

Storage: Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials.

Expiration: Due to mercury requirements, prepare this solution every 28 days or sooner as needed. The expiration date must not exceed the expiration date of any of the components.

- 7.3.3.3 ICP/MS Silver Spiking Solution – Add 10mL of hydrochloric acid to a 100-mL volumetric flask containing about 50mL of laboratory reagent water. Add the appropriate volume of

each single element stock standard to the flask. Dilute to volume with reagent water, and mix thoroughly. Transfer the spiking solution to a labeled storage container.

Element	Parent Standard Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Silver (Ag)	1000	2.5	500	5.0

## 8.0 Sample Collection, Preservation, Shipment, and Storage

### 8.1 Aqueous Samples

#### 8.1.1 Total Metals

Aqueous samples are routinely collected in 250mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

Note: Drinking water samples for the Lead and Copper Rule require 1 liter of sample to be collected.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

#### 8.1.2 Dissolved Metals

Aqueous samples for dissolved metals are routinely filtered at the time of sampling and collected in 500mL plastic containers containing 3mL of a 1:3 nitric acid preservative. The preservative should be sufficient to achieve a sample pH of less than 2.

Although no temperature preservation is required, samples are routinely iced at the time of collection at 0-6°C (less than 6°C but not frozen). Samples are stored at room temperature until the time of digestion. Samples must be digested and analyzed within 180 days of sample collection. If mercury is requested the samples must be digested and analyzed within 28 days of collection. Digestates are stored at room temperature until the time of analysis.

Note: If the sample is to be filtered in the laboratory, the sample must be collected in 500-mL plastic container with no preservatives. The sample must be stored at 0-6°C (less than 6°C but not frozen) until filtered. Once filtered, the laboratory will add nitric acid to obtain a pH of less than 2.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives.

#### 8.1.3 Silica Samples

Aqueous samples for silica are routinely collected in 500-mL plastic containers.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of filtration and analysis. Samples must be filtered and analyzed within 180 days of collection.

NCMs must be initiated for samples collected in improper containers.

Note: Acid preserved samples routinely collected for ICP analyses and acid digested samples should not be used for the analysis of silica. Also note that glass containers should be avoided.

#### 8.1.4 Preservation Checks – pH Verification

For each sample, prior to sample preparation,

- Place a piece of pH paper in a disposable medicine cup.
- Pour a few drops of sample into the medicine cup and note the color change of the pH paper.
- If the pH is outside the range of less than 2, initiate a Nonconformance Memo. Adjust the sample pH to less than 2 using 1:1 nitric acid.
- Mix well and hold for 24 hours. If pH is still greater than 2 after 24 hours, then repeat this process.

Note: To avoid cross-contamination, use a separate medicine cup and piece of pH paper per sample. Do not dip the pH paper into the sample container. The pH paper dye may bleed into the sample and affect sample results.

Note: Samples that are not at pH <2 upon arrival in the lab may contain cyanide or sulfide or may be highly buffered. Working under a hood minimizes the hazard that may be caused by the evolution of hydrogen cyanide or hydrogen sulfide upon acidification of the sample. Be aware that acid/base neutralization reaction may be violent and evolve a significant amount of heat.

#### 8.2 TCLP/SPLP Leachate Samples

Once the TCLP/SPLP extraction procedure has been performed, the leachate is transferred to a plastic container. The leachate must be acidified with nitric acid to pH <2, upon receipt to the Metals Department. The leachate sample must be digested and analyzed within 180 days of completion of the TCLP/SPLP extraction. If mercury is requested, the leachate sample must be digested and analyzed within 28 days of completion of the TCLP/SPLP extraction.

#### 9.0 Quality Control

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 of the associated analytical SOPs provide requirements for evaluating QC data.

## 9.1 Batch QC

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

### EPA 200.7 and EPA 200.8 – Drinking Water

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

### EPA 200.7 and EPA 200.8 – Clean Water Act

The laboratory's default minimum QC items performed for each digestion batch are: a method blank and a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the minimum QC items are a method blank, an LCS, 1 matrix spike, and a matrix spike duplicate.
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate.

### EPA Methods 3005A and 3010A and Standard Methods 3030C

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or a sample duplicate.

The routine container supplied for this method is a 250mL container. 50mL is required for extraction. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

If there is insufficient sample volume to perform the required matrix spike(s), the LCS must be prepared in duplicate (i.e., LCSD). An NCM must be initiated on all affected samples to denote this situation. Insufficient sample volume is defined as receiving less than a total of 100mL.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.



## 9.2 Instrument QC

Details on instrument QC are given in the associated analytical SOPs listed in Section 1.

### 9.2.1 Turbidimeter Instrument QC

Refer to SOP SA-GE-206 for additional information on the initial calibration of the turbidimeter. The Metals Department uses this turbidimeter only to assess whether drinking water samples can be analyzed undigested; therefore, a reduced QC frequency applied for this department.

The initial calibration curve must be verified initially and at the end of the analysis with low level Gelex standard (0-2NTU) standard. The CCV must be within 10% of the true value to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is the low level Gelex standard (0-2NTU) standard.

## 9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3 of the associated analytical SOP. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

## 10.0 Procedure

### 10.1 Sample and QC Sample Preparation

Unless otherwise requested, groundwater and surface waters are to be prepared using the total recoverable metals procedure given in Section 10.1.3. TCLP/SPLP samples must be digested for total metals (Section 10.1.4). Note that there are different spiking solutions for routine and TCLP/SPLP matrix spikes.

#### 10.1.1 Dissolved Metals

10.1.1.1 Results reported as “dissolved metals” are defined as the concentrations of analytes in a liquid sample that will pass through a 0.45um membrane filter prior to acidification/preservation. The filtration step can occur in the field or in the laboratory. In either case, the sample must be unpreserved prior to filtration and then filtered using a 0.45um filter.

10.1.1.2 Field filtered samples are usually preserved in the field, immediately after filtration. Unpreserved samples sent to the laboratory for lab filtration must be preserved immediately after filtration.

- 10.1.1.3 If the laboratory filters the samples, the reagent water used to prepare the method blank and laboratory control samples associated with the “dissolved metals” samples must be filtered through the same type of filter used to filter the field samples.

Note: The LCS should be spiked prior to filtration.

- 10.1.1.4 A comparison between total and dissolved results is good practice, and logic should follow that total results should be equal or greater than dissolved results, especially if the dissolved metals are lab filtered. Comparable hits with less than 20% RPD should be considered equal. Any other discrepancies should be discussed with the Department Manager or Supervisor, Technical Manager, or the Project Manager.

- 10.1.1.5 The laboratory’s standard practice is to digest all non-silica liquid samples; however, samples filtered for the determination of dissolved metals do not require digestion if the sample:
- has a low COD (<20mg/L);
  - has a turbidity <1 NTU;
  - is colorless with no significant odor; **and**
  - is of one liquid phase and free of suspended particulates or precipitates after acidification

#### 10.1.2 Turbidity Determination for Drinking Water Samples

Drinking water samples with a turbidity concentration of less than 1NTU may be analyzed without digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. The turbidity of drinking water samples is checked using the procedures outlined in this section. If the turbidity of the sample is not checked, the digestion procedure must be performed.

- 10.1.2.1 Turn on turbidimeter and allow it to warm up 15-30 minutes.
- 10.1.2.2 Verify the instrument is operating properly by the analysis of a continuing calibration verification sample initially, after every 25 samples, and at the end of the run with the lowest available Gelex standard (0-2). The Gelex standard must recover within  $\pm 10\%$  of the true value to be acceptable.
- 10.1.2.3 Analyze a DI water blank (CCB). The DI water blank must be less than 1NTU to be acceptable. If the DI water blank is greater the 1NTU, check to be certain the sample cell is clean. If the blank is significantly above 1NTU, the sample cell may be scratched and may need to be replaced.
- 10.1.2.4 Determine the turbidities for all samples by filling the cell with sample and placing the cell in the sample well of the turbidimeter. Record these turbidities in the Metals Turbidity Logbook. Be certain to rinse the cells between samples with DI water to avoid contamination.

#### 10.1.3 Total Recoverable Metals (EPA 3005A)

This digestion procedure is used to prepare aqueous samples for total recoverable metals determination by ICP and ICP/MS.

- 10.1.3.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

- 10.1.3.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

- 10.1.3.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

- 10.1.3.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

- 10.1.3.5 Add 2.5mL of concentrated HCl and 1.0mL of concentrated HNO<sub>3</sub> to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 15mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

- 10.1.3.6 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration must be limited to a factor of four.

10.1.3.7 The sample is now ready for analysis via the appropriate analytical SOP.

#### 10.1.4 Total Metals (EPA 3010, EPA 200.7, and EPA 200.8)

This digestion procedure is used for the preparation of aqueous samples for total metal determination by ICP and ICP/MS.

10.1.4.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.4.2 Add 50mL of reagent water to a digestion block vial to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.4.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water to serve as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010 EPA 200.7	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020 EPA 200.8	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.4.4 Add 0.50mL of the appropriate spiking solutions to two 50mL aliquots of the client sample designated as the matrix spike samples (MS and MSD).

10.1.4.5 Add 1.5mL of concentrated HNO<sub>3</sub> to each sample. Gently heat the digestion vessel until the sample refluxes. The sample must not be heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 5-10mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

- 10.1.4.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO<sub>3</sub>. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.
- 10.1.4.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO<sub>3</sub> until the digestate is light in color or does not change in appearance after subsequent additions of HNO<sub>3</sub>. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.
- 10.1.4.8 Evaporate the digestate until the volume is approximately 5-10mL.
- 10.1.4.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.
- 10.1.4.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.

Note: The digestate may be diluted to a volume less than the original volume if sample pre-concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

- 10.1.4.11 The sample is now ready for analysis via the appropriate analytical SOP.

#### 10.1.5 Total Metals and TCLP/SPLP Samples (EPA 3010)

This digestion procedure is used for the preparation of TCLP/SPLP leachate samples for total metal determination by ICP. Note that the LCS is spiked with the routine analytes to allow the TCLP/SPLP samples to be digested along with aqueous samples.

Note: The TCLP/SPLP leachate MS/MSD must be spiked prior to acidification. This is to be performed as close to 8 hours upon receipt of the leachate to the Metals Prep area as possible. Refer to Section 10.1.5.4 for more information.

- 10.1.5.1 Transfer a 5mL aliquot (diluted to 50mL) for TCLP or a 50mL aliquot for SPLP of a well-mixed sample to a clean 50mL digestion vessel. Larger volumes may be digested at the discretion of the lab. The volume of spike solution added should be adjusted proportionately.
- 10.1.5.2 Add 5mL of extraction fluid diluted to 50mL for TCLP or 50mL for SPLP to a digestion vessel to serve as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling. A blank for each type of extraction fluid must be digested and analyzed.
- 10.1.5.3 Add 0.50mL of the appropriate spiking solutions to a 5mL aliquot of extraction fluid diluted to 50mL for TCLP or to a 50mL aliquot of extraction fluid for SPLP. This is

designated as the laboratory control spike (LCS). If required, perform a LCSD by following the same procedure as the LCS. The routine ICP/MS spiking solution is used for the TCLP/SPLP LCS because of frequent client requests for reporting more analytes from the TCLP/SPLP leachate than are currently regulated. Preparing an LCS with all of the target analytes eliminates re-digestion and provides QC for the requested analytes.

Note: If both Extraction Fluid 1 and Extraction Fluid 2 are included in the batch, use Extraction Fluid 1 for the LCS and LCSD. Refer to SOP EX15: *Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP)* for information on the TCLP/SPLP extraction fluids.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010	TCLP/SPLP	LCS	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.5.4 The TCLP MS/MSD is prepared as follows: add 2.0mL of the ICPMS Calibration Stock and 0.4mL of the ICPMS Hg intermediate calibration standard to each of two 20mL aliquots of leachate.

The SPLP MS/MSD is prepared as follows: add 2.0mL of the ICPMS Calibration Stock and 0.04mL of the ICPMS Hg intermediate calibration standard to each of two 100mL aliquots of the leachate.

Note: The TCLP/SPLP digestion batch consists of twenty or fewer field samples and the associated QC items. A TCLP/SPLP digestion batch must not exceed 20 field samples. Every TCLP/SPLP digestion batch will have a method blank (MB), a laboratory control sample (LCS), and a matrix spike (MS).

10.1.5.5 Add 1.5mL of concentrated HNO<sub>3</sub> to each sample. Gently heat the digestion vessel until the sample refluxes. The sample is not heated to boiling; that is, bubbles are not formed in the liquid in the bottom of the digestion vessel. The sample/acid solution is refluxing when the liquid evaporates and drops of liquid condense on the sides of the digestion vessel and fall back into the digestion vessel. Evaporate the sample until the volume is approximately 5mL. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

10.1.5.6 Remove the digestion vessels from the digestion block and cool the digestion vessels to room temperature. Add another 1.5mL portion of concentrated HNO<sub>3</sub>. Continue heating the sample on the digestion block. Again, at the proper temperature, the sample should gently reflux in the digestion vessel. Do not allow the sample to boil.

10.1.5.7 Continue heating the sample and adding additional 1.5mL portions of concentrated HNO<sub>3</sub> until the digestate is light in color or does not change in appearance after subsequent additions of HNO<sub>3</sub>. If a sample requires more than 6mL of acid to digest, contact the Department Manager for guidance.



- 10.1.5.8 Evaporate the digestate until the volume is approximately 5-10mL.
- 10.1.5.9 Add 2.5mL of concentrated HCl and warm the sample digestate for 15 minutes.
- 10.1.5.10 Wash down the inside of the digestion vessel with reagent water. Dilute the sample digestate to 50mL with reagent water.
- 10.1.5.11 The sample is now ready for analysis by ICP.

#### 10.1.6 Acid-Extractable Metals (SM3030C)

This digestion procedure is used for the preparation of aqueous samples for acid-extractable metals by ICP and ICP/MS.

- 10.1.6.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a 50mL digestion block vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample, proportional volumes of reagents, and adjust the final digestate volume back to the original volume of the sample used. For example, if 25mL of sample is digested, one half of the routine volumes of reagent are used and the final volume of the digestate is brought back to 25mL. When a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

- 10.1.6.2 Add 50mL of reagent water to a beaker that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

- 10.1.6.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
EPA 6010	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
EPA 6020	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

- 10.1.6.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

- 10.1.6.5 Add 2.5mL of 1:1 HCl to each sample and QC item.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.



10.1.6.6 Heat for 15 minutes on a hot block.

10.1.6.7 The sample is now ready for filtration and final volume adjustment which occurs immediately prior to analysis by ICP or ICP/MS.

#### 10.1.7 Total Recoverable Metals (EPA 200.8 and EPA 200.7)

This digestion procedure is used for the preparation of water and drinking water samples for total recoverable metal determination by ICP and ICP/MS.

10.1.7.1 Transfer a 50mL aliquot (or an appropriate volume diluted to 50mL with reagent water) of a well-mixed sample to a clean 50mL block digestion vial.

Note: If there is not sufficient volume to use a 50mL aliquot, the lab can use a smaller volume of sample and bring the final digestate volume back to the original volume of the sample used. That is, if 25mL of sample is digested, the final volume of the digestate should be brought back to 25mL. If a smaller aliquot is used, the digestion analyst must be careful not to allow the sample digest to evaporate completely.

10.1.7.2 Add 50mL of reagent water to a block digestion vial that has been designated as the method blank. This QC sample is taken through all digestion and sample preparation steps to monitor for contamination that may be due to glassware, reagents, or sample handling.

10.1.7.3 Add 0.50mL of the appropriate spiking solutions to a 50mL aliquot of reagent water designated as the laboratory control spike (LCS). If a duplicate laboratory control spike (LCSD) is required, spike a second 50mL aliquot of reagent water with 0.50mL of the appropriate spiking solutions.

Analytical Method	Matrix	QC	Spiking Solutions
200.7 DW and TR	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike
200.8 DW and TR	Aqueous	LCS/MS/MSD	ICP/MS Spike 1 ICP/MS Spike 2 ICP/MS Ag Spike

10.1.7.4 Add 0.50mL of the appropriate spiking solutions to each of two 50mL aliquots of the client sample designated as the matrix spikes sample (MS and MSD).

10.1.7.5 Add 1.0 mL of (1:1) HNO<sub>3</sub> and 0.5 mL of (1:1) HCl to each sample. Gently heat the block digestion vial and reduce the sample volume to about 10mL without boiling; that is, bubbles are not formed in the liquid in the bottom of the block digestion vial. Do not allow any portion of the vessel bottom to become dry at any time during the digestion.

Note: If a volume of sample smaller than 50mL is digested, the amount of acid should be reduced proportionately.

- 10.1.7.6 Wash down the inside of the block digestion vial with reagent water. Dilute the sample digestate to 50mL with reagent water. Transfer the digest to a labeled storage container, usually a 125mL plastic vial. Allow any undissolved material to settle out or centrifuge the sample to remove particulate matter. A portion of the sample may be filtered, if necessary, to remove particulates but care must be taken to avoid contamination of the sample during filtration.

Note: The digestate may be diluted to a volume less than the original volume if sample concentration is required to meet lower reporting limits. The pre-concentration should be limited to a factor of four.

- 10.1.7.7 The sample is now ready for analysis by ICP and ICP/MS.

#### 10.1.8 Silica Samples

- 10.1.8.1 Samples are prepared by filtration of an unpreserved aliquot through a 0.45um syringe filter. The sample must be kept refrigerated until time of analysis. Note: The same number of syringe filters should be used for the method blank, LCS, and the samples.
- 10.1.8.2 The method blank is prepared by filtering an amount of reagent water equal to the volume of sample that is filtered.
- 10.1.8.3 The LCS is prepared by spiking 10mL of reagent water with 0.10mL of SiO<sub>2</sub> Intermediate Standard, and passing it through a 0.45um syringe filter. The SiO<sub>2</sub> spike concentration in the LCS is 10mg/L.
- 10.1.8.5 The matrix spike and/or matrix spike duplicate (MS/MSD) are prepared by adding 0.10mL of SiO<sub>2</sub> Intermediate Standard solution to two separate 10mL aliquots of the sample to be spiked and filtering through a 0.45um filter. The SiO<sub>2</sub> spike concentration in the MS/MSD is 10mg/L.

#### 10.2 Analysis

Details on sample analysis are given in the associated analytical SOPs listed in Section 1.

### 11.0 Calculations / Data Reduction

#### 11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

##### 11.1.1 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker feature to determine if historical data is available for review.

##### 11.1.2 Chemical Relationships

When available, the following chemical relationships must be evaluated for each sample. If these relationships are not met the Department Manager must be contacted immediately.

- Total results are  $\geq$  Dissolved results

## 11.2 Calculations

Details on sample calculations are given in the associated analytical SOPs listed in Section 1.

## 12.0 Method Performance

### 12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

### 12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

### 12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For analytes and methods certified by NELAC, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

#### 12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined annually, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

The IDL is defined as three times the standard deviation of seven replicate analyses of a blank solution analyzed over three non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in TALS.

#### 12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

#### 12.6 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

#### 12.7 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

### 13.0 **Pollution Control**

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

### 14.0 **Waste Management**

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

#### 14.1 **Waste Streams Produced by the Method**

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Acidic sample digestions: Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

## 15.0 **References / Cross-References**

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006: *Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Methods 3005A and 3010A: *Test Methods for Evaluating Solid Waste*, Third Edition, SW-846; vs. EPA Office of Solid Waste and Emergency Response: Washington, DC.
- Methods 200.7 and 200.8 (Drinking Water): *Methods for the Determination of Metals in Environmental Samples*, May 1994, Supplement 1. (EPA 600/R-94/111).
- *Standard Methods for the Examination of Water and Wastewater*, Online Edition; American Public Health Association: Washington, DC 2004.

## 16.0 **Method Modifications**

- 16.1 The EPA 200.7 and EPA 200.8 reference methods were written specifically for drinking water and source water samples; however, the laboratory may perform other types of water samples using this procedure.
- 16.2 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing an LCS at the RL in each EPA 200.7 and EPA 200.8 drinking water batch. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory defaults to qualitative identification of the LL-LCS to be acceptable.
- 16.3 There is no method-defined batch precision requirement for some of these methods. The EPA does require precision for all samples analyzed under the Clean Water Act, and the laboratory routinely performs an MSD to support clients who require precision to be reported. If insufficient sample volume is provided to perform the MS/MSD, the laboratory performs the LCS in duplicate (i.e., LCS/LCSD).
- 16.4 The turbidimeter is calibrated and maintained by the General Chemistry Department for use with turbidity analyses for EPA 180.1 and SM2130B. The Metals Department uses this turbidimeter only to assess whether drinking water samples can be analyzed undigested; therefore, a reduced QC frequency applied for this department. Refer to SOP SA-GE-206 for additional information on the calibration and verification of this meter.

## 17.0 **Attachments**

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

- Attachment 1: SOP Summary
- Attachment 2: Sample Collection, Preservation, and Holding Time Table
- Attachment 3: QC Summary
- Attachment 4: Instrument Maintenance and Troubleshooting
- Attachment 5: Turbidity Form
- Attachment 6: Glassware Cleaning Procedures



## **Attachment 1**

### **SOP Summary**

#### **Sample Preparation Summary**

**Total Metals and TCLP/SPLP leachates:** A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with nitric acid at approximately 95°C. After the sample has digested, as evidenced by a clear, pale yellow color, HCl is added and the sample is brought up to the original volume with reagent water. The laboratory utilizes two versions of the Total Recoverable preparation procedure. One version is equivalent to the EPA 3005A procedure and the second procedure is equivalent to the EPA 200.7 and 200.8 procedures.

**Total Recoverable Metals:** A known volume, usually 50mL, of sample is transferred to a digestion vessel. The sample is refluxed with dilute nitric acid and hydrochloric acid at approximately 95°C. After the sample has evaporated to approximately 10-20mL, the sample is brought up to the original volume with reagent water. This procedure is equivalent to EPA Method 3005A, and the EPA Methods 200.7 and 200.8 prep procedures for total recoverable metals.

**Acid Extractable Metals:** A known volume, usually 50mL of sample preserved with nitric acid, is transferred to a digestion vessel. Hydrochloric acid is added and the sample is heated on a hot block for 15 minutes. The sample is filtered and the final volume adjusted to the original volume. This procedure is equivalent to Standard Methods 3030C.

**Silica samples for EPA Methods 200.7 and 6010C:** Liquid samples are filtered. The filtered liquid samples are analyzed by ICP.

**Drinking water samples for EPA Methods 200.7 and 200.8** with a turbidity concentration of less than 1NTU may be analyzed with no digestion. The exception to this rule is silver, which requires sample digestion prior to analysis. If the sample turbidity is >1NTU, then the EPA Method 200.7 preparation procedure or the EPA Method 200.8 preparation procedure is used.

Samples filtered for the determination of dissolved metals do not require digestion if the sample:

- 5) has a low COD(<20mg/L);
- 6) has a turbidity <1NTU ;
- 7) is colorless with no significant odor ; **and**
- 8) is of one liquid phase and free of suspended particulates or precipitates after acidification.

#### **Sample Analysis Summary**

**ICP:** Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT). The PMTs, located behind the exit

slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

ICP/MS: Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

### **Analytical Sequence**

See the appropriate analytical SOP for information on the analytical sequence.

ICP: SOP SA-ME-070: *Elements by ICP*

ICP/MS: SOP SA-ME-074: *Elements by ICP/MS*

**Attachment 2:  
Sample Collection, Preservation, and Holding Time Table**

Matrix	Routine Sample Container	Routine Sample Size	Minimum Sample Size	Dechlorination Agent	Chemical Preservation	Thermal Preservation	Holding Time <sup>2</sup>
Water	250mL Plastic	50mL	25mL	Not Applicable	1:1 HNO <sub>3</sub> to pH<2	Not Applicable	Mercury: 28 days from collection  Other Metals: 180 days from collection
Water (Lead & Copper Rule Only)	1L Plastic	50mL	25mL	Not Applicable	1:1 HNO <sub>3</sub> to pH<2	Not Applicable	180 days from collection
Water (Silica)	250mL Plastic	50mL	25mL	Not Applicable	Not Applicable	0-6°C <sup>1</sup>	180 days from collection
Leachate	100mL or 250mL Plastic	50mL	25mL	Not Applicable	1:1 HNO <sub>3</sub> to pH<2	Not Applicable	Mercury: 28 days from leaching  Other Metals: 180 days from leaching  Note: SM3030C has a 72 hour HT.

<sup>1</sup> Samples are collected on ice and maintained at <6°C with no frozen samples.

<sup>2</sup> Inclusive of digestion and analysis.

Note: If dissolved metals are requested, the sample must be filtered prior to the acid being added to the sample.

### Attachment 3: QC Summary

QC Item	Frequency	Criteria	Corrective Action
Batch Definition	Digested together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Lab Control Sample (LCS)	One per batch	Refer to analytical SOP	Refer to analytical SOP
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	TALS MLG	Redigest and reanalyze batch
Low-Level Laboratory Control Sample (LLCS) – spiked at the RL	EPA 200.7 and EPA 200.8 DW: One per batch	Qualitatively identified	If the “regular” LCS meets criteria, initiate NCM and report data If the “regular” LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7 and EPA 200.8: 10% of samples prepared; i.e., 2 separate matrix spikes per batch of twenty samples  Other Methods: 5% of samples prepared; i.e., 1 matrix spike per batch of twenty samples	TALS MLG	Flag and report data
Matrix Spike Duplicate (MSD) or Sample Duplicate (SD)	One per batch	TALS MLG	Flag and report data
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006  Note: Unsupervised work must not begin until acceptable IDOC is

QC Item	Frequency	Criteria	Corrective Action
			obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

## **Attachment 4: Instrument Maintenance and Troubleshooting**

### **Instrument Labeling**

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

### **Maintenance Log**

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

### **Preventive Maintenance**

Refer to the instrument manufacturer's guides for trouble-shooting items.

The temperature of the hot plate or digestion block must be monitored with each batch. If the temperature required for sample preparation cannot be maintained, the heating device must be removed from service and repaired or replaced.

### **Troubleshooting**

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

### **Contingency Plan**

Maintenance contracts are carried for most instrumentation and close contact is maintained with service personnel to ensure optimal instrument functioning. An extensive spare parts inventory is maintained for routine repairs, consisting of thermocouples, digestion tubes, and turbidimeter cells, and other common instrumentation components. Since instrumentation is standardized throughout the laboratory network, spare parts and components can be readily exchanged among the network.

In general, the laboratory has at least one backup unit for each critical unit. In the event of instrument failure, portions of the sample load may be diverted to duplicate instrumentation, the analytical technique switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples shipped to another properly certified or approved TestAmerica location.

## Attachment 5: Turbidity Form

### Turbidity for Metals Drinking Water Samples

**Date:**  
**Analyst Initials:**  
**CCV LIMS Standard Number:**  
**CCV True Value:** NTU

CCV criteria:  $\pm 10\%$  of True Value  
CCB criteria:  $< 1$  NTU  
If sample turbidity is  $< 1$  NTU and silver is not requested, the sample does not require digestion.

[illegible]



## Attachment 6: Glassware Cleaning Procedures

GLASSWARE CLEANING PROCEDURES METALS DEPARTMENT	
<p style="text-align: center;"><b>Graduated Cylinders</b></p> <ol style="list-style-type: none"> <li>1. Scrub with hot, soapy H<sub>2</sub>O and brush.</li> <li>2. Rinse thoroughly with tap H<sub>2</sub>O.</li> <li>3. Rinse with 10% HNO<sub>3</sub></li> <li>4. Rinse thoroughly with DI H<sub>2</sub>O.</li> </ol>	
<p style="text-align: center;"><b>Volumetric Flasks</b></p> <ol style="list-style-type: none"> <li>1. Empty contents of flask</li> <li>2. Squirt a small amount of cleaning detergent directly into volumetric flask</li> <li>3. Fill flask 1/3 full with HOT H<sub>2</sub>O</li> <li>4. Replace top and shake flask.</li> <li>5. Empty flask and rinse with HOT H<sub>2</sub>O until no soap remains in flask.</li> <li>6. Add approximately 10mL concentrated HNO<sub>3</sub> to 50mL, 100mL, and 250mL flasks — replace top and shake well. For 500mL or 1000mL flasks use 25mL and for 10mL flasks use 2 - 5mL of concentrated HNO<sub>3</sub>.</li> <li>7. Rinse 3 times with DI H<sub>2</sub>O, filling flask 1/3 full and replacing top. Store until needed.</li> </ol> <p><b>* NEVER PLACE VOLUMETRIC FLASKS OR TOPS IN SINK OR DISHPAN WITH OTHER DIRTY DISHES.</b></p>	
<p><b>*Dispose of all acid waste in accordance with the TestAmerica Savannah Addendum to the Corporate Safety Manual.</b></p>	

FME023:03.11.09:3



## 18.0 Revision History

### Summary of Changes from Previous Revision:

- Minor editorial and formatting changes made.
  - o Updated SOP references to reflect current document control number designations.
  - o Replaced reference to LIMS with TALS.
- Added reference to Safety Data Sheets/SDS. Section 5.2.
- Added requirement to scan/attach standard COAs to TALS. Section 7.3
- Added ICP/MS Silver Spiking Solution to Standards section. Section 7.3.3.3
- Adjusted sample collection and storage conditions to reflect 0-6°C. Section 8.1 and Attachment 2
- Replaced detail regarding initial calibration of turbidimeter with reference to SA-GE-206 as this is performed by the General Chemistry department, who regularly conduct EPA Method 180.1 and SM 2130B. Removed Turbidimeter ICAL Form. Section 9.2.1 and Section 16.4
- Changed required frequency of turbidimeter CCV to beginning and end of each sequence. Section 9.2.1.3
- Added ICP/MS Ag Spike to list of spiking solutions to include in LCS/MS/MSD. Section 10.1
- Removed requirements to filter and to adjust final volume of sample for acid extractable metals, as these steps are performed at the bench prior to analysis. Sections 2.3 and Section 10.1.6
- Added Volumetric Container table. Section 6.0
- Added Glassware Cleaning Procedures. Attachment 7
- Added requirement to collect samples for Lead and Copper Rule using 1L containers. Section 6.4, Section 8.1.1, and Attachment 2


## ELEMENTS BY ICP


(Methods: EPA 200.7, EPA 6010C, SM2340B)

### Approvals (Signature/Date):

 \_\_\_\_\_ April 16, 2014  
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Quality Assurance Manager  
Date

 \_\_\_\_\_ April 3, 2014  
Whitney Palefsky  
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## 1.0 **Scope and Application**

This SOP gives the procedures for the determination of metals (elements) by inductively coupled plasma (ICP) atomic emission spectroscopy.

The routine matrices for this procedure are waters and soils; however, this procedure may be adapted to accommodate other matrices as outlined in Section 16.1.

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the LIMS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

## 2.0 **Summary of Method**

Prior to analysis by ICP, the sample must be digested/filtered using the sample preparation method appropriate to the analyte/matrix combination. Sample digestates/filtrates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or onto a charge-coupled device (CCD). The PMTs and CCDs, located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

Note: Drinking water samples (EPA 200.7) only require digestion if the determination of silver (Ag) is requested or if the turbidity is greater than or equal to 1.0 NTU.

This SOP is based on the following methods: EPA 200.7 and EPA 6010C. The procedure for the determination hardness via the calculation method (i.e., SM2340B) is provided in Attachment 9.

## 3.0 **Definitions**

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

## 4.0 **Interferences**

### 4.1 **Procedural Interferences**

- 4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.

- 4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in accordance with the posted Labware Cleaning Instructions to ensure it is free from contaminants and does not contribute artifacts.
- 4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.
- 4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.
- 4.1.5 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. Typically, physical interferences are mitigated by the instrument's peristaltic pump and the use of internal standard.

#### 4.2 Matrix Interferences

- 4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix. The sample may require dilution prior to analysis to reduce or eliminate the interferences.
- 4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.
- 4.2.3 Spectral interferences are caused by the overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous phenomena, and stray light from the line emissions of highly concentrated elements.
  - 4.2.3.1 Spectral overlap may be compensated for by the use of inter-element correction factors.
  - 4.2.3.2 Background contribution and stray light can be compensated for by a background correction adjacent to the analyte line.

#### 5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the

responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable latex or nitrile gloves, and closed-toe, nonabsorbent shoes when handling samples.

#### 5.1 Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

The plasma generates high temperatures. Analysts must ensure that all equipment is shut down and cooled off before performing maintenance and troubleshooting in the plasma area.

Nitric and hydrochloric acids are extremely hazardous as oxidizers, corrosives, poisons, and are reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose, throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, and pain. Nitric acid can cause deep ulcers, and staining of the skin to a yellow or yellow-brown color. These acid vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage.

Samples that contain high concentrations of carbonates or organic matter, or samples that are at elevated pH can react violently when acids are added. Acids must be added to samples under a hood to avoid splash/splatter hazards and/or possibly toxic vapors that will be given off when the samples are acidified.

#### 5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

**NOTE: This list does not include all materials used in the procedure.** A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the "MSDS" link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit <sup>1</sup>	Signs and Symptoms of Exposure
Hydrochloric Acid <sup>2</sup>	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid <sup>2</sup>	Corrosive Oxidizer Poison	2ppm TWA  4ppm STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
<sup>1</sup> Exposure limit refers to the OSHA regulatory exposure limit.			
<sup>2</sup> Always add acid to water to prevent violent reactions.			

## 6.0 Equipment and Supplies

### 6.1 Equipment and Instrumentation

Varian 730 ES or other suitable inductively coupled plasma emission spectrometer. Data is evaluated and quantitated using TALS

Top-loading Balance – Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

### 6.2 Lab Supplies

Argon gas supply and appropriate fittings

Nitrogen Gas

Cooling water supply

Peristaltic pump

Volumetric Containers – various sizes; Class A, where applicable. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*



Pump-style Pipettes – various sizes. Verify in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*

pH paper

### 6.3 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers provided by the laboratory are as follows:

Waters: 250mL Plastic – purchased with Certificate of Analysis attesting to purity.

Solids: 8oz Plastic or Glass Jar – purchased with Certificate of Analysis attesting to purity.

## 7.0 Reagents and Standards

The standards and reagents listed are those in use at the time this SOP was updated. Other standard and reagent stocks, vendors, and concentrations may be used provided they are fully documented and in compliance with method requirements.

### 7.1 Expiration Dates

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life – i.e., when in doubt, throw it out.

The expiration date of any standard must not exceed the expiration date of the standard that was used to prepare it; that is, the "children may not outlive the parents".

Unless listed elsewhere in this SOP, the expiration dates given below apply.

- 7.1.1 The expiration date for unopened standards and reagents is the manufacturer's expiration date.
- 7.1.2 The expiration date for opened stock reagents is the manufacturer's expiration date or 5 years from the date opened, whichever is sooner.
- 7.1.3 The expiration date for opened stock standards is the manufacturer's expiration date.
- 7.1.4 The expiration date for prepared reagents is 6 months from the date prepared or the expiration date of the parent reagent, whichever is sooner.
- 7.1.5 The expiration date for prepared standards is 6 months from the date prepared or the expiration date of the parent standard, whichever is sooner.

### 7.2 Reagents

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*.

Hydrochloric acid and nitric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

- 7.2.1 Laboratory Reagent Water – ASTM Type I. The conductivity must be monitored in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*.
- 7.2.2 Nitric acid (HNO<sub>3</sub>) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.
- 7.2.3 Hydrochloric acid (HCl) – reagent grade. Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Store away from sunlight, heat, water, and incompatible materials. Stable under ordinary conditions of use and storage.

### 7.3 Standards

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. Certificates of analysis or purity must be received with all purchased standards, and scanned and filed in the Data Archival Folder on the Public\_QA-drive.

Refer to Attachments 5 and 6 for standard preparation information.

## 8.0 Sample Collection, Preservation, Shipment, and Storage

### 8.1 Aqueous Samples

Aqueous samples are routinely collected in 250mL plastic containers containing 1.5mL 1:1 nitric acid preservative or 500mL plastic containers containing 3.0mL 1:1 nitric acid preservative. The preservative must be sufficient to achieve a sample pH of less than 2.

If dissolved metals are requested, the sample must be filtered in the field. This filtration step must occur prior to the addition of the preservative.

If the laboratory is requested to filter the sample, then the sample must be collected in a 250mL plastic container without preservative. The samples are filtered upon receipt and are then acidified with 1.25mL concentrated nitric acid to a pH<2. Note: Samples must sit for 24 hours after the addition of the acid to verify pH is maintained, prior to digestion.

Note: The reference methods state that filtration must occur immediately; therefore, if the laboratory is requested to filter the samples – as opposed to a field filtration - appropriate narration must be included in the final report to denote this method deviation.

Samples are stored at room temperature until the time of digestion. Samples must be

digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

## 8.2 Soil Samples

Soil samples are routinely collected in 8oz plastic or glass containers.

Samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until the time of digestion. Samples must be digested within 6 months of collection. Digestates are stored at room temperature until the time of analysis and analyzed within 6 months of collection.

## 9.0 Quality Control

SOP SA-QA-017: *Analytical Batching and Evaluation of QC Data* and the SOP Summary in Attachment 4 provide requirements for evaluating QC data.

### 9.1 Batch QC

#### 9.1.1 EPA 200.7 – Drinking Water

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a low-level LCS (LLCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS, an LLCS, 1 matrix spike, and 1 matrix spike duplicate (MSD).
- For a batch of 11-20 samples, the minimum QC items are a method blank, an LCS, an LLCS, 1 matrix spike (from sample 1-10), another matrix spike (from sample 11-20), and a matrix spike duplicate (MSD).

#### 9.1.2 EPA 200.7 – Clean Water Act

A digestion batch consists of up to 20 environmental samples and the associated QC items digested together within a 24 hour period.

The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS) to be performed on a minimum of 10% of samples or one per batch – whichever is greater, and a matrix spike duplicate (MSD).

This frequency equates to the following:

- For a batch of 10 or fewer samples, the default QC items are a method blank, an LCS,

1 MS, and 1 MSD.

- For a batch of 11-20 samples, the default QC items are a method blank, an LCS, 1 MS (from sample 1-10), another MS (from sample 11-20), and an MSD.

#### 9.1.3 EPA 6010C

A digestion batch consists of up to 20 environmental samples and the associated QC items. The laboratory's default minimum QC items performed for each digestion batch are: a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD).

- 9.1.4 If there is insufficient sample provided to perform the required matrix spikes, the LCS must be prepared induplicate (i.e., LCS/LCSD). An NCM must be initiated to denote this situation.

Insufficient sample volume is defined as receiving less than a total of 50mL or 2g.

The routine container supplied for this method is a 250mL (water) or 8oz (soil) container. 50mL or 1g is required for digestion. Reduced sample initial volumes may be necessary to achieve the required batch matrix spike frequency; however, the minimum extraction volume to be used for the matrix spike samples is 25mL or 0.5g. Note: Final volumes and spike amounts must be adjusted to compensate for these reduced initial volumes.

- 9.1.5 Batch QC must meet the criteria given in Attachment 3 of this SOP.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

#### 9.1.6 Preparation SOPs

Refer to the following SOPs for specifics on the preparation process:

Matrix	SOP
Water Samples	SA-ME-050
Soil Samples	SA-ME-051

### 9.2 Instrument QC

#### 9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of at least one standard (the high standard) and a blank. If a multi-point curve is requested, the calibration curve must consist of a minimum of 3 standards and a blank. The initial calibration standard concentrations currently in use in the laboratory for a multi-point curve are included in Attachment 5.

Note: Refer to Attachment 5 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

The correlation coefficient (r) of the regression curve must be greater than or equal to 0.998 for the initial calibration curve to be acceptable.

If a multi-point calibration curve is used, the upper limit of the linear range is the concentration of the High Standard.

#### 9.2.1.1 Determination of Linear Range of the ICP

If the instrument is not calibrated over its entire linear range for a particular element, a linear range standard must be analyzed immediately following the calibration to validate the linear range.

This standard must recover within +/- 10% of the true value and any sample that is greater than 90% of the concentration in the linear range standard must be diluted and reanalyzed.

#### 9.2.2 High Standard Read Back

The highest concentration calibration standard must be reanalyzed as an "unknown" after the instrument is calibrated. The results for the re-analysis of the highest concentration calibration standard must be within  $\pm 5\%$  of the true value for each target analyte. If the result for any target analyte is outside of this range, the ICP may need to be "profiled" and the standardization/calibration repeated.

#### 9.2.3 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified initially – prior to any sample analyses – in accordance with SOP SA-QA-016 with a standard obtained from a second source.

The ICV must be within 10% of the true value to be acceptable for EPA 6010C. The ICV must be within 5% of the true value to be acceptable for EPA 200.7, and must also have a %RSD  $\leq 3\%$  of the 3 replicates analyzed to be acceptable for EPA 200.7.

The initial calibration verification standard concentration currently in use in the laboratory is given in Attachment 5. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

#### 9.2.4 Initial Calibration Blank (ICB) / Continuing Calibration Blank (CCB)

The instrument must be shown to be free from contamination by the analysis of calibration blanks. Initial calibration blanks are analyzed immediately following the initial calibration. Continuing calibration blanks must be analyzed immediately following each continuing calibration verification (CCV).

The absolute value of the initial and continuing calibration blanks must be  $< \frac{1}{2}RL$  to be acceptable.

#### 9.2.5 Continuing Calibration Verification

The initial calibration curve must be verified every 10 samples with a mid-level standard.

The CCV must be within 10% of the true value to be acceptable and must also have a %RSD  $\leq 5\%$  of the 3 replicates.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to half the concentration of the High Standard. Refer to Attachment 5 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

#### 9.2.6 Internal Standard (ISTD)

This procedure is an internal standard (ISTD) procedure. Yttrium is used as the primary internal standard; however, Scandium may also be used as an alternate ISTD.

The internal standard must be added to all standards, samples, and QC items prior to analysis. This is accomplished by means of an additional channel on the peristaltic pump and connected to the sample line with a 'T' or 'Y' connector fitting. This ensures constant concentration of the internal standard and eliminates the possibility of human spiking error. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples. A concentration of 7mg/L is used for Yttrium and Scandium.

Any sample containing ISTD recoveries greater than 130% must be diluted and re-analyzed. Although ISTD recoveries less than 70% are extremely rare, the analyst should consider further dilution if this situation occurs.

##### 9.2.6.1 Ionization Effects

High concentrations of some elements, such as Na, K, Ca, and Mg, can produce interferences from their ionization effects. The introduction of additional ions in abundance, such as Lithium, will minimize this interference. Therefore, Lithium is added to the internal standard solution, and is continually pumped at a constant rate.

See Attachment 5 for details on the preparation of the internal standard solution.

#### 9.2.7 Reporting Limit Check Standard

A reporting limit (RL) check standard is used to demonstrate that the ICP is capable of detecting the target compounds at or below the reporting limit. EPA 200.7 requires this check only when utilizing a single point calibration. EPA 6010C requires this check for both single point and multi-point calibrations.

The concentrations in the RL check standard must be at levels that are less than or equal to the reporting limit for the samples being analyzed. The determined concentration must be detected within  $\pm 50\%$  (for EPA 200.7) or  $\pm 30\%$  (for EPA 6010C) of the true concentration. The RL check standard must be analyzed at the beginning and end of an analysis run (i.e., all samples must be bracketed by this check standard).

Note: This RL Check Standard is referred to as the LLICV and LLCCV in EPA 6010C.

#### 9.2.8 Interference Check Standards

The purpose of the Interference Check Standard is to prove that the instrument software is adequately correcting for common interferences through the use of interelement correction factors. The concentrations of the target analytes must be within 20% of the true concentrations to be acceptable. The analyst must pay particular attention to false positives and false negatives for elements not present in the interference check solutions.

#### 9.2.9 Serial Dilution

A dilution must be prepared and analyzed on one sample per batch to determine if matrix interferences are present. Compare the results of the diluted and un-diluted aliquots of sample digestate for analytes that are present in the native sample at a concentration  $\geq 50 \times$  IDL.

If the results of the dilution are within  $\pm 10\%$  of the results of the undiluted sample, no matrix interference is present. If the results differ by greater than the amounts outlined above, a matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

#### 9.2.10 Post-Digestion Spike

A post-digestion spike is performed on one sample per analytical batch to determine if matrix interferences are present. This post-digestion spike is evaluated if the serial dilution fails or if the analyte concentration is not at least 50 times the instrument detection limit. The sample selected as the post-digestion spike should be the same sample selected for serial dilution.

The post-digestion spike must be within 25% of the true value to be acceptable. If these criteria are not met, then the data are qualified.

#### 9.2.11 % RSD of Multiple Exposures

Replicate analyses of a sample should agree within 30% RSD for samples with detections greater than the reporting limit. Replicates that are greater than 30% RSD should be investigated for matrix interferences or differences in viscosity of the digest.

#### 9.2.12 Interelement Correction Factors (IEC)

Interelement correction factors (IEC) for all elements must be determined every six months using the manufacturer's guidance.

The laboratory may combine the linearity study with the IEC study, thereby eliminating redundancy. (Single element linearity check solutions for each analyte should be closely evaluated for all non-spiked elements, and applicable correction factors should be applied according to the instrument's software. The analyst must be careful not to correct for any contamination which may be present in the actual solution.)



At a minimum the IECs must be verified at the beginning of each analytical sequence through the analysis of interferent check solutions ICSA and ICSAB. Note: Some protocols and project plans require the analysis of the ICSA and ICSAB standard at the beginning and end of an analysis sequence. The analyst must be aware of the requirements of the project before beginning the analysis.

### 9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

## 10.0 Procedure

### 10.1 Sample Preparation

The sample preparation procedures are given in the following SOPs:

Matrix	SOP #
Water Samples	SA-ME-050
Soil Samples	SA-ME-051

### 10.2 QC Sample Preparation

Batch QC sample preparation procedures are given in the SOPs listed above. Additional QC items are listed below.

#### 10.2.1 Serial Dilution

Dilute the digestate by a factor of five, and analyze the dilution using the same procedures used for the un-diluted aliquot.

#### 10.2.2 Post Digestion Spike

Transfer 10mL of a digestate to a suitable vial. Spike the sample with 0.10mL of Spike II.

### 10.3 Analysis

All soil digestates and all water digestates containing particulates are filtered through a 0.45um filter prior to analysis.

Note: If digestates are filtered prior to analysis, then the applicable method blank and LCS/LCSD must also be filtered. This information must be captured via the Batch Data Types in TALS.

#### 10.3.1 Instrument Operating Conditions

Turn the ICP on and allow it to become thermally stable before beginning to analyze the calibration standards. It will take about an hour for the instrument to warm up. If the optics were turned off, allow 2 hours warm up time. After the warm-up period, perform a torch alignment sequence to properly profile the instrument.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

#### 10.3.2 Internal Standard (ISTD)

Prior to analysis, internal standard must be added to all standards, samples, and QC items. The concentration of the internal standard must be the same in all calibration samples, field samples, and QC samples.

#### 10.3.3 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described in Section 9.2.1. Once the calibration has been established and verified with a high level standard and an ICV in accordance with Sections 9.2.2 and 9.2.3, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.5.

#### 10.3.4 Sample Analysis

Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the complete removal of trace analytes. The instrument blanks would be included in the sample count in determining the maximum number of samples between CCVs/CCBs.

The default procedure is to include QC items (method blank, LCS, MS/MSD, and SD) in determining the maximum number of samples between CCVs/CCBs.

#### 10.3.5 Example Analytical Sequence

An example analytical sequence is listed below.

Analytical Sequence for samples immediately following an initial calibration:

Description	Comments
Instrument Warm-up	
Torch Alignment	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Reporting Limit Check Standard	

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

## 11.0 Calculations / Data Reduction

### 11.1 Data Reduction

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

#### 11.1.1 Dilutions

If the concentration of a sample is above the calibration range (or 90% of the linear range for single point curves) of the instrument the sample digestate must be diluted and reanalyzed.

Dilutions must be prepared in reagent water containing 5% hydrochloric acid and 1% nitric acid by volume.

#### 11.1.2 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, the analyst must verify TALS Worksheet Notes and/or use the TALS Historical Data Tracker feature to determine if historical data is available for review.

### 11.1.3 Chemical Relationships

The analyst must be aware of the following chemical relationships:

- Total Results should be  $\geq$  Dissolved results

If this relationship is not met, further evaluation is needed to rule out a laboratory error.

### 11.1.4 Evaluation of Batch QC and Samples for Matrix Interferences

Matrix interference is evaluated using three criteria: the recoveries of the matrix spike (MS) and matrix spike duplicate (MSD), the results of the serial dilution, and the recovery of the post digestion spike. This laboratory uses internal standard calibration and quantification which minimizes the need to use the method of standard additions (MSA) to quantify sample digests with known or suspect matrix interferences. MSA is used when the recoveries of the MS/MSD are less than 50% and there is no obvious reason for the poor recoveries (e.g., viscosity of digest, high dissolved solids, undigested solids, known high concentrations of inter-element interferences, etc.).

The primary indicator of matrix interference is the results of the MS/MSD. If the recoveries and precision of the MS/MSD are within acceptance limits, matrix interferences are not indicated and unacceptable results for the serial dilution and post-digestion spikes are likely due to poor preparation rather than matrix interference.

Use the following table to evaluate for the indication of matrix interferences.

MS	MSD	Serial dilution <sup>1</sup>	Post Digestion Spike <sup>1</sup>	Action
OK	OK	OK	OK	Report
OK	OK	Out	OK	Unlikely to occur; suspect preparation of serial dilution or post digestion spike
OK	OK	OK	Out	
OK	Out	OK	OK	Suspect preparation-qualify results
Out	OK	OK	OK	
Out	Out	Out	OK	Matrix interference confirmed-qualify results if recoveries >50%
Out	Out	OK	Out	
Out	Out	Out	Out	If MS/MSD recoveries <50% and there is no obvious reason, perform MSA as directed in section 11.1.5.

<sup>1</sup>Performed on un-spiked digest of the sample designated as the MS/MSD

#### 11.1.5 Evaluation of Post-Digestion Spikes

Result of Post-Digestion Spikes	Action
Within 75-125% limits	None
>125% recovery	Re-spike and reanalyze if multiple analytes >125%. Repeat analysis of un-spiked sample and re-evaluate recoveries. Qualify results for affected analytes.
<75% recovery but >50% recovery	Qualify results for affected analytes.  Dilute digestate and repeat spike for affected analytes. <b><i>Treat all samples associated with spike in the same manner as the spiked sample (i.e., spike or dilute samples).</i></b> If recoveries are not 75-125%, all associated samples may be re-analyzed by single point MSA.  Note: High levels of target analytes may inhibit spike recovery. Consult the Department Manager in events where high levels of targets appear to be interfering.
<50% recovery	Dilute and re-spike. Elevate RL accordingly (for all associated samples). Spike and evaluate all associated samples. Spike and evaluate all associated samples by single point MSA. Qualify results for affected analytes.

Note: The >50% recovery of the post digestion spike is a benchmark below which samples may be biased high if corrected for spike recovery.

#### 11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD and % difference).

11.2.2 The calculations associated with initial and continuing calibrations are given in SOP SA-QA-016. Applicable calculations include determination for correlation coefficient, slope, intercept, and percent recoveries.

11.2.3 The calculation to determine final concentration is given as follows:

$$FinalConcentration = CONC_{Sample} \otimes \frac{F}{I \times dw} \otimes D$$

Where:

CONC<sub>Sample</sub> = Concentration of the sample  
F = Final volume/weight  
I = Initial volume/weight  
D = Dilution factor  
dw = % Solids decimal equivalent

Note: All dry weight corrections are performed automatically in LIMS.

**Note: This calculation assumes all applicable unit correction factors are applied.**

#### 11.2.4 Method of Standard Additions

The concentrations of both sample aliquots are measured and the sample concentration is calculated as follows:

$$C_x = \frac{S_2 V_s C_s}{(S_1 - S_2) V_x}$$

Where:

S<sub>1</sub> = Absorbance or concentration of the spiked aliquot  
S<sub>2</sub> = Absorbance or concentration of the un-spiked aliquot  
V<sub>s</sub> = Volume of spike solution  
V<sub>x</sub> = Volume of sample aliquots  
C<sub>s</sub> = Spike solution concentration

**Note: This calculation assumes all applicable unit correction factors are applied.**

### 12.0 Method Performance

#### 12.1 Reporting Limit Verification (RLV)

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

#### 12.2 Method Detection Limit (MDL) Study

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

#### 12.3 Method Detection Limit Verification (MDLV)

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

#### 12.4 Determination of the Instrument Detection Limit (IDL)

The instrument detection limit (IDL) is the concentration of analyte that can be statistically distinguished from the background noise of the instrument. The IDL limit must be determined quarterly, at a minimum, for each analyte in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits (RLs, MDLs, and IDLs)*.

The IDL is defined as three times the average of the standard deviation of a minimum of seven replicate analyses of blank solution performed three times over non-consecutive days. The IDL may be elevated above the background noise (blank levels). The current IDL associated with this procedure is given in the Equipment Limit Group (ELG) in TALS.

#### 12.5 QC Limit Generation, Control Charting, and Trend Analysis

The control limits for the batch QC items (LCS, MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

#### 12.6 Lower Limit of Quantitation Check Sample (LLQC)

EPA 6010C requires a lower limit of quantitation check sample (LLQC) analysis to demonstrate the required reporting limit capability. The laboratory shall prepare and analyze this solution on a yearly basis, in accordance with all applicable preparatory SOPs (i.e. the solution must be digested.) It is recommended that the laboratory utilize the RL Check solution (Section 9.2.7) by simply carrying it through the entire preparatory



procedure for each of the prep methods associated with EPA 6010C. Digesting this solution on the same prep batch as the yearly MDL studies is preferred. The LLQC must recover within 70-130% to be acceptable, or a new (higher) reporting limit must be established.

#### 12.7 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

#### 12.8 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs.

### 13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (EHSM) and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

### 14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the

TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

#### 14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.
- Excess soil and solid samples – Dispose according to characterization on sample disposal sheets. Transfer non-hazardous samples to TCLP container for characterization in hazardous waste department. Transfer hazardous samples (identified on disposal sheets) to waste department for disposal.
- Acidic sample digestions – Neutralize before disposal into drain/sewer system.

Dispose of all metals standards with concentrations exceeding the TCLP threshold limits as a RCRA aqueous hazardous waste. The TCLP constituents and the respective threshold limits are:

Analyte	Disposal Limit (mg/L)
Arsenic	5.0
Barium	100
Cadmium	1.0
Chromium	5.0
Lead	5.0
Selenium	1.0
Silver	5.0
Mercury	0.20

Neutralize the standard by passing with sodium bicarbonate before transferring the waste to the RCRA aqueous hazardous waste drum.

All other metals standards may be disposed, using copious amounts of water, down a sink that flows to the neutralization pit.

#### 15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Procedures*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006: *Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-015: *Homogenization, Compositing, and Segregation of Samples*

- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- *Methods for Chemical Analysis of Water and Waste*; U.S EPA Office of Research and Development: Cincinnati, OHIO, March 1983.
- EPA 200.7, Revision 4.4, EMMC Version: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry; 1994
- *Methods for the Determination of Metals in Environmental Samples*; US EPA Office of Research and Development. Washington, DC.
- *Test Methods for Evaluating Solid Waste, Third Edition*; U.S. EPA Office of Solid Waste and Emergency Response: Washington, D.C., November 1986 (SW-846 IV).
- Method 6010C, Revision 3: Inductively Coupled Plasma-Atomic Emission Spectrometry; February 2007.
- *Standard Methods for the Examination of Water and Wastewater, Online Edition*; American Public Health Association: Washington, DC.
  - SM2340B: Hardness by Calculation (Approved 1997, Editorial Revision 2011)

## **16.0 Method Modifications/Clarifications**

### **16.1 Incorporation of Non-Routine Matrices**

This procedure may be modified to analyze other matrices (e.g., wipe, waste, tissue, filter, and TCLP/SPLP leachate samples) upon client request. This will need to be arranged by the Project Manager at the initiation of the project.

Wipe, waste, filter, and tissue matrices are non-routine, and the laboratory is not currently NELAC certified for these matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate wipe, waste, filter, and tissue samples. Soil DOCs can be used to satisfy analyst demonstrations of capability for these types of non-routine matrices. The laboratory uses its routine soil RLs (converted for initial and final volumes, etc.) and soil QC limits to evaluate TCLP/SPLP leachate samples. Water DOCs can be used to satisfy analyst demonstrations of capability for TCLP/SPLP matrices. Teflon chips, Ottawa sand, or equivalent is used as the blank matrix for wipes, wastes, filters, and tissues unless specifically requested otherwise by the project.

#### **16.1.1 Collection and Handling Procedures for Non-Routine Matrices**

Waste (oil) samples are collected in 500mL plastic containers. Waste (oil) samples are stored at room temperature until the time of digestion. Wipe samples are collected in 20mL scintillation vials. Wipe and filter samples must be iced at the time of collection and maintained at 4°C (less than 6°C but not frozen) until time of digestion. Waste (oil), wipe, and filter samples must be digested within 6 months of collection.

Tissue samples are collected in plastic containers with the size dependent upon the type of tissue being collected. Plastic jars or plastic baggies can be used. Upon receipt, tissue

samples must be placed in the freezer at -10° to -20°C for up to 6 months if digestion cannot be completed that day. Tissue samples must be digested within 6 months of thawing.

Digestates for waste (oil), wipe, tissue, and filter samples are stored at room temperature until the time of analysis and must be analyzed within 6 months of collection.

Once the TCLP/SPLP extraction procedure has been performed, the TCLP/SPLP leachate must be transferred to a 500mL plastic container and preserved with 1.0mL nitric acid to a pH <2. Preserved TCLP/SPLP leachates are stored at room temperature until the time of digestion. The leachate sample must be digested within 6 months of completion of the TCLP/SPLP extraction. Digestates are stored at room temperature until the time of analysis and must be analyzed within 6 months of completion of the TCLP/SPLP extraction.

#### 16.1.2 Preparation and Analytical Procedures for Non-Routine Matrices

Wipe, waste, filter, and tissue samples are prepared in the same manner as routine soil samples as outlined in SOP SA-ME-051. TCLP/SPLP matrices are prepared in the same manner as routine water samples as outlined in SOP SA-ME-050. Refer to the applicable preparation SOPs for more information.

Wipe, waste, filter, tissue, and TCLP/SPLP matrices are analyzed in the same manner as routine samples as outlined in this SOP.

#### 16.2 Other Considerations

16.2.1 The EPA Manual for the Certification of Laboratories Analyzing Drinking Water requires a LFB at the MRL to be performed each day. The laboratory meets this requirement by preparing a low-level LCS (LLCS), spiked at the RL, in each EPA 200.7 batch of drinking water samples. The EPA DW Manual does not specify criteria for the low-level LCS; therefore, the laboratory requires this standard to be qualitatively identified to be acceptable.

16.2.2 The stock standards utilized for the Silica procedure are routinely purchased as Silicon (Si). Both Silica (SiO<sub>2</sub>) and Silicon (Si) can be reported using this procedure. The following equation is used to convert between Silicon (Si) and Silica (SiO<sub>2</sub>):

$$Si = \frac{SiO_2}{2.14}$$

16.2.3 The laboratory's default QC items incorporate an MSD to satisfy the Clean Water Act requirements and those clients who batch require precision to be reported. Additionally, if insufficient sample volume is provided to perform the MS/MSD, the LCS is routinely prepared in duplicate (i.e., LCS/LCSD).

16.2.4 Linear Range determinations are performed initially upon instrument set-up and when significant instrument changes are made. Linear Range verifications are performed quarterly as required by DOD QSM. This frequency is greater than that defined by the reference methods.

- 16.2.5 EPA Method 200.7 requires that IEC studies be performed annually. The laboratory defaults to a 6-month frequency. This frequency is greater than that defined by the reference method and is consistent with the requirement outlined in EPA Method 6010C.
- 16.2.6 The reference methods do not specify a %RSD criteria for replicate exposures of the CCV. The laboratory has incorporated an RSD criteria of 5% for all CCVs, as required by the DOD QSM.
- 16.2.7 The reference methods require IDLs to be performed annually. The laboratory performs IDLs quarterly as required by the DOD QSM.
- 16.2.8 The reference methods state that, for dissolved metals analysis, filtration must occur in the field. The laboratory may be requested to filter the samples upon receipt. If this occurs, then appropriate narration must be included in the final report to denote this method deviation.

**17.0 Attachments**

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

- Attachment 1: SOP Summary
- Attachment 2: Sample Collection, Preservation, and Holding Time Table
- Attachment 3: QC Summary
- Attachment 4: Instrument Maintenance and Troubleshooting
- Attachment 5: Standard Preparation Tables
- Attachment 6: Silica/Silicon Standard Preparation
- Attachment 7: Linear Range Determination
- Attachment 8: Element Wavelengths
- Attachment 9: Hardness Calculation Work Instruction

## Attachment 1: SOP Summary

### Sample Preparation Summary

Prior to analysis by ICP, the sample must be digested using the sample preparation method appropriate to the matrix. Samples should be prepared according to the appropriate matrix-specific SOP.

Matrix	SOP #
Aqueous Samples	SA-ME-050
Soil Samples	SA-ME-051

### Sample Analysis Summary

Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. As the elements fall to a lower energy level, radiation characteristic of the elements present in the plasma is emitted. The light is directed through an entrance slit, dispersed by the diffraction grating, and projected on to the photomultiplier tube (PMT) or on to a charge-coupled device (CCD). The PMTs and CCDs located behind the exit slits, convert the light energy to an electrical current. This signal is then digitized and processed by the data system. Background correction is required for trace element determination.

### Analytical Sequence

Description	Comments
Instrument Warm-up	
Torch Alignment	If torch is changed
Initial Calibration	
High Calibration Standard	Re-analyzed as a sample
ICV	Second Source
ICB	
Reporting Limit Check Standard	
ICP Interference Check Solution A (ICSA)	
ICP Interference Check Solution AB (ICSAB)	
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Samples & Batch QC Items	Up to 10 analyses, including QC
CCV	
CCB	10-analysis count begins after analysis of the CCB
Reporting Limit Check Standard	Method criterion

Note: The analytical sequence continues as outlined above and must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

**Attachment 2:**  
**Sample Collection, Preservation, and Holding Time Table**

<b>Matrix</b>	<b>Routine Sample Container</b>	<b>Routine Sample Size</b>	<b>Minimum Sample Size</b>	<b>Dechlorination Agent</b>	<b>Thermal Preservation<sup>1</sup></b>	<b>Chemical Preservation<sup>1</sup></b>	<b>Holding Time<sup>2</sup></b>
Water	250mL or 500mL plastic	50mL	25mL	Not Applicable	None required	1:1 Nitric Acid to pH<2	6 months
Water (Silica)	250mL or 500mL plastic	10mL	5mL	Not Applicable	4°C <sup>3</sup>	Not Applicable	6 months
Soil	8oz plastic or glass	1g	0.50g	Not Applicable	4°C <sup>3</sup>	Not Applicable	6 months
Soil (Silica)	8oz plastic or glass	5g	2.5g	Not Applicable	4°C <sup>3</sup>	Not Applicable	6 months

<sup>1</sup>Samples requiring dissolved metals must be filtered prior to preservation.

<sup>2</sup>Inclusive of digestion and analysis.

<sup>3</sup>Samples must be collected on ice and maintained at 0-6°C with no frozen samples.



**Attachment 3:  
QC Summary**

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Daily	Single Point: 1 standard and 1 blank  Multi-point: Minimum 3 standards and 1 blank Correlation coefficient $\geq 0.998$	Recalibrate
High Standard	Immediately after every calibration	Recoveries within $\pm 5\%$ of expected values	New initial calibration
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	EPA 6010C: within $\pm 10\%$  EPA 200.7: within $\pm 5\%$ , $\leq 3\%RSD$	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within $\pm 10\%$ of true value $\leq 5\%RSD$	Terminate the analysis, correct the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value $< \frac{1}{2}RL$	Terminate the analysis, correct problem and reanalyze the previous 10 samples
Interference Check Standards (ICSA/ICSAB)	At the beginning of an analysis run	$\pm 20\%$ of the true values Pay attention to false positives and false negatives for elements not present in the solutions.	Terminate the analysis, correct the problem, recalibrate, and reanalyze all samples since the last ICS that was in control.
Batch Definition	Up to 20 field samples digested together within a 24-hr time period	Not Applicable	Not Applicable
Method Blank (MB)	One per batch of twenty samples or less	$ result  < \frac{1}{2}RL$ or result $< 10\%$ of the analyte level in the sample	Refer to SOP SA-QA-017
Laboratory Control Sample	One per batch	TALS MLG	Refer to SOP SA-QA-017

QC Item	Frequency	Criteria	Corrective Action
(LCS)			
Laboratory Control Sample Duplicate (LCSD)	One per batch, if insufficient sample provided for MS/MSD	TALS MLG	Refer to SOP SA-QA-017
Low-Level Laboratory Control Sample (LLCS)  – at the RL	EPA 200.7 DW Only: One per batch of twenty samples or less for drinking water	Qualitatively detected	If the “regular” LCS meets criteria, initiate NCM and report data If the “regular” LCS does not meet criteria, redigest and reanalyze batch
Matrix Spike (MS)	EPA 200.7: 10% of samples prepared; i.e., 2 separate matrix spikes per batch  EPA 6010C: 5% of samples prepared; i.e., 1 matrix spike per batch	TALS MLG	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	One per batch	TALS MLG	Refer to SOP SA-QA-017
Serial Dilution (1/5 Dilution)	One per batch	Refer to Section 9.2.9	Refer to Section 9.2.9
Post Digestion Spike (PDS)	One per batch	Refer to Section 9.2.10	Refer to Section 9.2.10
Reporting Limit Check Solution (also known as LLICV and LLCCV for EPA 6010C)	At the beginning and end of an analysis run (Required for EPA 200.7 when using a single point calibration. Required for EPA 6010C when using both single point and multi-point calibrations.)	EPA 200.7: +/-50% of the true concentration  EPA 6010C: +/-30% of the true concentration	Opening: Stop the analysis, fix the problem, and reanalyze the affected samples.  Capping: Qualify affected samples; reanalyze if required by project plan

QC Item	Frequency	Criteria	Corrective Action
%RSD (CV) of Multiple Exposures	Evaluate for all Calibration, QC, and samples	Conc. $\geq$ RL - Warning: $\leq 20\%$ - Acceptance: $\leq 30\%$	-if CV $>20$ but $\leq 30$ , review data for possible interferences; -if interference present, reanalyze digest -if no interference present, report average -if CV $\geq 30\%$ , reanalyze digest, report the result that has the lower precision value or dilute the digestion and reanalyze
		Conc. $<$ RL -use professional judgment	-use professional judgment
Linear Range	Determined at least every 6 months in accordance with Attachment 7	% difference $\leq 10\%$	-reanalyze at a lower concentration
Interelement Correction Factors (IEC)	Determined every 6 months. Verified at the beginning of an analysis run	Refer to ICSA, ICSAB criteria	See ICSA, ICSAB corrective action
Lower Limit of Quantitation Check Sample	Annually	EPA 6010C: 70-130%	- reanalyze at a higher concentration - elevate RL accordingly
Initial Demonstration of Capability (IDOC)	Initially, per analyst, per analyte/method/matrix combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006 Note: Unsupervised work must not begin until acceptable IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually, per analyst, per analyte/method combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

QC Item	Frequency	Criteria	Corrective Action
	DOD ELAP) or annually thereafter (for non-DOD ELAP)		
Method Detection Limit Study (MDL)	Upon method/instrument set-up, per analyte/method/matrix combination	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for non-DOD ELAP)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Instrument Detection Limit (IDL)	Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007

## Attachment 4: Preventative Maintenance and Troubleshooting

### Preventive Maintenance

EQUIPMENT ITEM	Service Interval							SERVICE LEVEL
	D	W	M	Q	SA	A	AN	
ICAP Service Schedule								
Pump Tubing							X	Inspect and replace as needed (Recommend daily)
Nebulizer							X	Clean as needed
Filters							X	Clean or replace as needed (Recommend monthly)
Chiller Water Filter							X	Clean or replace as needed (Recommend every six months)
Injector Tip / Torch							X	Clean or replace as needed (Recommend Daily inspection)
Tubing Connectors							X	Replace as needed

D = daily; W = Weekly; M = monthly; Q = Quarterly; SA = semi-annually; A = annually;  
AN = as needed

### Troubleshooting

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

### Maintenance Log

A maintenance log must be established for each piece of equipment used in the laboratory. All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

### Instrument Labeling

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

**Attachment 5:  
Standard Preparation**

**Note: All standards must be stored at room temperature and have an expiration date of 6 months from date prepared.**

**Continuing Calibration Verification (CCV)**

**Final Volume (mL):** 2000

**Solvent:** 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Single Element Stocks**

Ag	1000	CPI	1.0	0.50
Al	10000	SPEX	1.0	5.0
B	1000	CPI	10.0	5.0
Fe	10000	CPI	1.0	5.0
Sr	1000	ABSOLUTE	5.0	2.5
Ti	1000	CPI	1.0	0.50
Na	10000	CPI	0.50	2.5
Sb	1000	CPI	1	0.5

**Multi Element Mixes**

**CAL STD 2**

As	50	CPI	20	0.50
Mo	50			0.50
Pb	50			0.50
Sb	50			0.50
Se	500			5.0
Tl	500			5.0

**CAL STD 3**

Ba	500	CPI	20	5.0
Be	50			0.50
Cd	50			0.50
Co	50			0.50
Cr	500			5.0
Cu	500			5.0
Mn	500			5.0
Ni	250			2.5
Zn	250			2.5

**CAL STD 5**

Ca	500	CPI	20	5.0
K	1000			10
Mg	500			5.0
Na	500			5.0
Sn	500			5.0

V	500			5.0
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**Internal Standard (ISTD)**

**Final Volume (mL):** 10000

**Solvent:** 30% HNO<sub>3</sub>

<b>Purchased Standards</b>	<b>Concentration (mg/L)</b>	<b>Vendor *</b>	<b>Amount Used</b>	<b>Final Concentration (mg/L)</b>
Y	10000	CPI	7.0mL	7.0
Lithium Carbonate (powder)	18.78% Li	Mallinckrodt	5.0g	94.65
Sc	10000	CPI	7.0mL	7.0



**MDL & IDL Intermediate**

**Final Volume:**

**(mL)** 100

**Solvent:** 5% HCL / 1 % HNO<sub>3</sub>

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Single Element Stocks**

Ag	1000	CPI	0.040	0.40
Al	10000	SPEX	0.10	10
As	1000	CPI	0.20	2.0
B	1000	CPI	0.40	4.0
Ba	1000	CPI	0.020	0.20
Be	1000	CPI	0.0050	0.10
Ca	10000	SPEX	0.10	10
Cd	1000	CPI	0.040	0.40
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.10	1.0
Fe	10000	CPI	0.10	10
K	10000	CPI	0.20	20
Mg	10000	SPEX	0.10	10
Mn	1000	CPI	0.020	0.20
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	2.04	204
Ni	1000	CPI	0.10	1.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.20	2.0
Sr	1000	CPI	0.040	0.40
Ti	1000	CPI	0.050	0.50
Tl	1000	CPI	0.20	2.0
V	1000	CPI	0.060	0.60
Zn	1000	CPI	0.10	1.0

**SPIKE II – Post Spike**

**Final Volume**

**(mL):** 500

**Solvent:** 5% HCL / 1 % HNO<sub>3</sub>

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Single Element Stocks**

Ag	1000	CPI	2.5	5.0
Al	10000	SPEX	10	200
As	1000	CPI	10	20
Ba	1000	CPI	10	20
B	1000	CPI	25	50
Be	10000	CPI	2.5	5.0
Ca	10000	SPEX	10	200
Cd	1000	CPI	2.5	5
Co	1000	CPI	10	20
Cr	1000	CPI	10	20
Cu	10000	CPI	10	20
Fe	10000	CPI	10	200
K	10000	CPI	10	200
Mg	10000	SPEX	10	200
Mn	1000	CPI	10	20
Mo	1000	CPI	10	20
Na	10000	CPI	10	200
Ni	1000	CPI	10	20
Pb	1000	CPI	10	20
Sb	1000	CPI	10	20
Se	1000	CPI	10	20
Sn	1000	CPI	10	20
Sr	1000	CPI	10	20
Ti	1000	CPI	10	20
Tl	1000	CPI	10	20
V	1000	CPI	10	20
Zn	1000	CPI	10	20

**ICP-MS LCS/MATRIX SPIKE SOLUTION**

Preparation of the ICP-MS Matrix Spiking / Post Digestion Spiking Solution

Element	Parent Concentration (mg/L)	Volume Added (mL)	Final Volume (mL)	Final Concentration (mg/L)
Aluminum (Al)	10000	5.0	100	500
Antimony (Sb)	1000	0.5		5
Arsenic (As)	1000	1.0		10
Boron (B)	1000	2.0		20
Barium (Ba)	1000	1.0		10

Beryllium (Be)	1000	0.5		5.0
Cadmium (Cd)	1000	0.5		5.0
Calcium (Ca)	10000	5.0		500
Cobalt (Co)	1000	1.0		5.0
Chromium (Cr)	1000	1.0		10
Copper (Cu)	1000	1.0		10
Iron (Fe)	10000	5.0		500
Lead (Pb)	1000	0.50		5.0
Magnesium (Mg)	10000	5.0		500
Manganese (Mn)	1000	5.0		50
Mercury (Hg)	1000	0.050		0.50
Molybdenum (Mo)	1000	1.0		10
Nickel (Ni)	1000	1.0		10
Potassium (K)	10000	5.0		500
Selenium (Se)	1000	1.0		10
Silver (Ag)	1000	0.50		5.0
Sodium (Na)	10000	5.0		500
Strontium (Sr)	1000	1.0		10
Thallium (Tl)	1000	0.4		4.0
Tin (Sn)	1000	1.0		10
Titanium (Ti)	1000	1.0		10
Vanadium (V)	1000	1.0		10
Zinc (Zn)	1000	1.0		10

Separate mixtures can be utilized for stability considerations.

#### RL ICP INTERMEDIATE

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO<sub>3</sub>

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
<b>Single Element Stocks</b>				
Ag	1000	CPI	0.10	1.0
Al	10000	SPEX	0.20	20
As	1000	CPI	0.20	2.0
B	1000	CPI	1.0	10
Ba	1000	CPI	0.10	1.0
Be	1000	CPI	0.040	0.4
Ca	10000	SPEX	0.50	50
Cd	1000	CPI	0.050	0.5
Co	1000	CPI	0.10	1.0
Cr	1000	CPI	0.10	1.0
Cu	1000	CPI	0.20	2.0
Fe	10000	CPI	0.050	5.0

K	10000	CPI	1.0	100
Mg	10000	SPEX	0.50	50
Mn	1000	CPI	0.10	1.0
Mo	1000	CPI	0.10	1.0
Na	10000	CPI	1.0	100
Ni	1000	CPI	0.40	4.0
Pb	1000	CPI	0.10	1.0
Sb	1000	CPI	0.20	2.0
Se	1000	CPI	0.20	2.0
Sn	1000	CPI	0.50	5.0
Sr	1000	CPI	0.10	1.0
Ti	1000	CPI	0.10	1.0
Tl	1000	CPI	0.25	2.5
V	1000	CPI	0.10	1.0
Zn	1000	CPI	0.20	2.0

**RL ICP Working**

**Final Volume (mL):** 1000

**Solvent:** 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**From RL ICP Intermediate**

Ag	1.0	CPI	10.0	0.010
Al	20	SPEX		0.20
As	2.0	CPI		0.020
B	10	CPI		0.10
Ba	1.0	CPI		0.010
Be	0.4	CPI		0.0040
Ca	50	SPEX		0.50
Cd	0.5	CPI		0.0050
Co	1.0	CPI		0.010
Cr	1.0	CPI		0.010
Cu	2.0	CPI		0.020
Fe	5.0	CPI		0.050
K	100	CPI		1.0
Mg	50	SPEX		0.50
Mn	1.0	CPI		0.010
Mo	1.0	CPI		0.010
Na	100	CPI		1.0
Ni	4.0	CPI		0.040
Pb	1.0	CPI		0.010
Sb	2.0	CPI		0.020
Se	2.0	CPI		0.020
Sn	5.0	CPI		0.050
Sr	1.0	CPI		0.010
Ti	1.0	CPI		0.010
Tl	2.5	CPI		0.025
V	1.0	CPI		0.010
Zn	2.0	CPI		0.020

ICV (different  
vendor used for ICV)

Final Volume (mL): 100

Solvent: 5% HCL / 1 % HNO<sub>3</sub>

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Single Element Stocks**

Na	1000	Ultra	0.09	9.0
Sn	1000	Ultra	0.50	5.0
Sr	1000	Ultra	0.05	5.0

**Multi Element Stocks**

Ultra26				
As	100	Ultra	1.0	1.0
Be	100			1.0
Ca	100			1.0
Cd	100			1.0
Co	100			1.0
Cr	100			1.0
Cu	100			1.0
Fe	100			1.0
Mg	100			1.0
Mn	100			1.0
Mo	100			1.0
Ni	100			1.0
Pb	100			1.0
Sb	100			1.0
Se	100			1.0
Tl	100			1.0
V	100			1.0
Zn	100			1.0

Ultra 26 cont				
Ag	100	Ultra	5.0	1.0
Al	100			1.0
B	100			1.0
Ba	100			1.0
K	1000			10
Na	100			1.0
Si	50			0.50

**ICSA**

**Final Volume (mL):** 1000

**Solvent:** 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Multi Element Mixes**

<u><b>Interferents A</b></u>				
Al	5000	SPEX	100	500
Ca	5000			500
Mg	5000			500
Fe	2000			200



**ICSAB**

**Final Volume (mL):** 1000

**Solvent:** 5% HCL / 1 % HNO3

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Multi Element Mixes**

Interferents A				
Al	5000	SPEX	100	500
Ca	5000			500
Mg	5000			500
Fe	2000			200

Altany C				
Ag	20	SPEX	10	0.20
As	10			0.10
Ba	50			0.50
Be	50			0.50
Cd	100			1.0
Co	50			0.50
Cr	50			0.50
Cu	50			0.50
Mn	50			0.50
Ni	100			1.0
Pb	5			0.050
Sb	60			0.60
Se	5			0.050
Tl	10			0.10
V	50			0.50
Zn	100			1.0

Trace AB				
Mo	100	CPI	10	1.0
Sn	100			1.0

**Trace AB**

**Final Volume (mL):** 100

**Solvent:** 5% HCL / 1 % HNO<sub>3</sub>

<b>Purchased Standards</b>	<b>Concentration (mg/L)</b>	<b>Vendor *</b>	<b>Volume Used (mL)</b>	<b>Final Concentration (mg/L)</b>
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**Single Element Stocks**

Mo	1000	CPI	10	100
Sn	1000	CPI	10	100

**High Std**

**Final Volume (mL):** 1000

**Solvent:** 5% HCL / 1 % HNO<sub>3</sub>

Purchased Standards	Concentration (mg/L)	Vendor *	Volume Used (mL)	Final Concentration (mg/L)
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**Single Element Stocks**

Ag	1000	CPI	1.0	1.0
Al	10000	SPEX	1.0	10
B	1000	CPI	10	10
Fe	10000	CPI	1.0	10
Na	10000	CPI	0.50	5.0
Sr	1000	ABSOLUTE	5.0	5.0
Ti	1000	CPI	1.0	1.0
Sb	1000	CPI	1.0	1.0

**Multi Element Mixes**

CAL STD 2				
As	50	CPI	20	1.0
Mo	50			1.0
Pb	50			1.0
Sb	50			1.0
Se	500			10
Tl	500			10

CAL STD 3				
Ba	500	CPI	20	10
Be	50			1.0
Cd	50			1.0
Co	50			1.0
Cr	500			10
Cu	500			10
Mn	500			10
Ni	250			5.0
Zn	250			5.0

CAL STD 5				
Ca	500	CPI	20	10
K	1000			20
Mg	500			10
Na	500			10
Sn	500			10
V	500			10

## Attachment 6: Silica/Silicon Standard Preparation

Silicon (Si) stock solutions are usually purchased for this procedure. The following conversion is used to adjust any volumes or concentrations appropriately:

$$Si = \frac{SiO_2}{2.14}$$

- Stock SiO<sub>2</sub> Standard, 10000mg/L Si / 21400mg/L SiO<sub>2</sub> – purchased from CPI. Store at room temperature. This standard must be used by the manufacturer's expiration date.
- Stock Second Source SiO<sub>2</sub> Initial Calibration Verification (ICV), 50mg/L Si / 107mg/L SiO<sub>2</sub> QC7 Standard, purchased from Ultra. Store at room temperature. This standard must be used by the manufacturer's expiration date.

Note: The ICV must be prepared from a stock standard that is obtained from a different source than the stock standard used to prepare the calibration standard. The second source must be from a separate vendor, unless a second vendor is unavailable, in which case a separate lot from the same vendor may be used.

- Intermediate SiO<sub>2</sub> Standard, 467mg/L Si / 1000mg/L SiO<sub>2</sub> – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the Stock SiO<sub>2</sub> Standard given in the table below to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. Stock SiO <sub>2</sub> Std (mg/L)	Volume Stock SiO <sub>2</sub> Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> ) [Silicon (Si)]	21400 [10000]	4.67	100	1000 [467]

- Initial Calibration Standards
  - Preparation of the Calibration Blank (ICB, CCB) – The calibration blank is reagent water. The calibration blank is used as the initial calibration blank (ICB) and the continuing calibration blank (CCB).
  - High Level SiO<sub>2</sub> Calibration Standard, 10mg/L SiO<sub>2</sub> – Add 20mL to 30mL of reagent water to a clean, plastic 100-mL volumetric flask. Add the volume of the Intermediate SiO<sub>2</sub> Standard given in the table, below, to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO <sub>2</sub> Intermediate Std (mg/L)	Volume SiO <sub>2</sub> Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> )	1000	1.0	100	10

- Mid Level SiO<sub>2</sub> Calibration Standard (also used as CCV solution), 5.0mg/L SiO<sub>2</sub> – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO <sub>2</sub> Intermediate Std (mg/L)	Volume SiO <sub>2</sub> Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> )	1000	0.50	100	5

- Low Level SiO<sub>2</sub> Calibration Standard (also used as RL solution), 0.50mg/L – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table, below, to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. SiO <sub>2</sub> Intermediate Std (mg/L)	Volume SiO <sub>2</sub> Intermediate Std (mL)	Final Volume (mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> )	1000	0.050	100	0.50

- Second Source SiO<sub>2</sub> ICV Standard, 0.50mg/L SiO<sub>2</sub> / 1.07mg/L Si – Add 20mL to 30mL of reagent water to a clean, plastic 100mL volumetric flask. Add the volume of the stock standard given in the table to the volumetric flask. Dilute to volume with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Element	Conc. of Stock Std (currently from Element) (mg/L)	Volume Stock Std (mL)	Final Volume of Cal Std mL)	Final Conc. (mg/L)
Silica (SiO <sub>2</sub> ) [Silicon (Si) ]	107 [ 50 ]	1.0	100	1.07 [ 0.50 ]

- Stock ICP Interference A Check Standard – purchased Interference A Stock Standard, from SPEX. Store the standard at room temperature. This standard must be used by its manufacturer's expiration date.

Note: This solution does not contain any  $\text{SiO}_2$ . Concentrations are as follows:

Aluminum (Al)	5000mg/L
Calcium (Ca)	5000mg/L
Magnesium (Mg)	5000mg/L
Iron (Fe)	2000mg/L

- ICP Interference A Check Standard, Working – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of the Stock ICP Interference A Check Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Note: This solution does not contain any  $\text{SiO}_2$ . Final concentrations are as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L

- ICP Interference Check Solution AB – Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Stock ICP Interference A Check Standard and 0.50mL of the Intermediate  $\text{SiO}_2$  Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

The final concentrations of this solution should be as follows:

Aluminum (Al)	500mg/L
Calcium (Ca)	500mg/L
Magnesium (Mg)	500mg/L
Iron (Fe)	200mg/L
Silica ( $\text{SiO}_2$ )	5.0mg/L

- Linearity Check Solution, 100mg/L – P Add 20mL to 30mL of reagent water to a clean 100-mL plastic volumetric flask. Add 10mL of Intermediate  $\text{SiO}_2$  Standard to the volumetric flask. Dilute to a final volume of 100mL with reagent water. Store the standard at room temperature. This standard must be used by its parent standard's expiration date or within 6 months of preparation, whichever comes first.

Note: Historically, the Linear Dynamic Range for silica has been 100mg/L; however, the instrument is dynamic. The concentration of this standard may need to be adjusted based how the instrument is performing. Prepare other volumes and/or concentrations according to the following equation:

$$V_s = \frac{V_{lc} \otimes C_{lc}}{C_s}$$

where

$V_s$  = volume of stock standard (mL)

$C_s$  = concentration of stock standard (mg/L)

$V_{lc}$  = volume of linearity check standard to prepare (mL)

$C_{lc}$  = concentration of linearity check standard to prepare (mg/L)



## Attachment 7:

### Linear Dynamic Range Determination and Verification

#### Initial Linear Dynamic Range Determination:

The linear dynamic range (LDR) of each instrument must be determined initially, upon instrument set-up, and whenever significant instrument changes are made following the procedure outlined below:

- Prepare and analyze individual standards at concentrations (a minimum of three are recommended) that are expected to define the linear dynamic range of the instrument.

Note: The calibration standards and the LDR standards must be matrix matched; that is, they must have the same percentage of hydrochloric and nitric acids.

- Compare the concentration of each standard with its true concentration as follows:

$$PercentDifference = \left| \frac{C_{cal} - C_{true}}{C_{true}} \right| \otimes 100$$

Where:

$C_{cal}$  = concentration determined from analysis

$C_{true}$  = true concentration of the standard

- Continue analyzing increasing concentrations of standards (recommend increasing by a factor of two) until the percent difference is greater than 10%. The highest standard that meets the 10% criteria establishes the initial LDR of the instrument.

Note: The laboratory routinely utilizes a “working” LDR that is equivalent to the concentration of the High Standard of the calibration curve. This working LDR allows for consistency between instruments and must not be greater than the LDR established by the initial LDR study.

#### Linear Dynamic Range Verification:

The working LDR must be verified quarterly by analyzing a standard at this concentration. If the percent difference is less than or equal to 10%, the working LDR is confirmed at that concentration. If the percent difference is greater than 10%, repeat the initial LDR Study as outlined above.

#### Evaluation of Samples with Relation to the Linear Dynamic Range:

Samples with concentrations greater than 90% of the working LDR must be diluted to bring the concentration below 90% of the working LDR.

## Attachment 8: Element Wavelengths

Element	Wavelength ICP-D (nm)	Wavelength ICP-E (nm)
Aluminum (Al)	308.215	308.215
Antimony (Sb)	206.838	206.834
Arsenic (As)	189.042	188.98
Barium (Ba)	493.409	389.178
Beryllium (Be)	313.042	313.042
Boron (B)	249.678	249.678
Cadmium (Cd)	226.502	226.502
Calcium (Ca)	317.933	370.602
Chromium (Cr)	267.716	267.716
Cobalt (Co)	228.616	228.615
Copper (Cu)	324.753	324.754
Iron (Fe)	271.441	271.441
Lead (Pb)	220.353	220.353
Magnesium (Mg)	279.078	279.078
Manganese (Mn)	257.610	257.610
Molybdenum (Mo)	202.030	202.032
Nickel (Ni)	231.604	231.604
Potassium (K)	766.491	766.491
Selenium (Se)	196.026	196.026
Silver (Ag)	328.068	328.068
Sodium (Na)	330.231	330.237
Strontium (Sr)	421.542	216.596
Thallium (Tl)	190.864	190.794
Tin (Sn)	189.989	189.925
Titanium (W)	334.941	334.941
Vanadium (V)	292.402	292.401
Zinc (Zn)	206.200	206.200

Note: Other wavelengths may be used with DM and TM approval.

## Attachment 9: Hardness Calculation Work Instruction



**Work Instruction**  
FME157:05.23.07.0  
Page 1 of 1

### **CALCULATION OF HARDNESS**

(as Calcium Carbonate)

Method: SM2340B

#### **Summary of Procedure:**

An aqueous sample is analyzed for calcium and magnesium using ICP by methods EPA 200.7 or 6010B (SOP ME70: *Elements by ICP*), or using ICP/MS by methods EPA 200.8 or 6020 (SOP ME74: *Elements by ICP/MS*). The concentrations of these elements are converted to equivalents of calcium carbonate using the equation below and added together to give the final result.

#### **Calculation:**

**Determine the concentration of calcium and magnesium as described in SOP ME70 or ME74.**

**Calculate the hardness as calcium carbonate using the following equation:**

$$\text{Hardness (as mg/L calcium carbonate)} = (4.118 \times [\text{Mg}]) + (2.497 \times [\text{Ca}])$$

where:

[Mg] = Concentration of magnesium (mg/L)

[Ca] = Concentration of calcium (mg/L)

#### **Quality Control:**

Report the method blank as the default QC unless otherwise requested.

Formal detection limit studies, as described in 40CFR Part 136B and SOP QA07: *Determination of Detection Limits*, are not required for SM2340B.

This procedure is a calculation based on another analytical procedure; therefore, demonstration of capability (DOC) is accomplished by successful completion of analytical DOCs. (i.e. If the analyst has a successful DOC on file for Ca and Mg by EPA 200.7/6010B or EPA 200.8/6020, proficiency has been shown for SM2340B by default.)

## 18.0 Revision History

### Summary of Changes from Previous Revision:

- Minor editorial, grammatical, and/or formatting changes made. Updated SOP numbers to reflect current document control designations. Replaced reference to G-Drive with Public\_QA-Drive. Replaced references to LIMS with TALS.
- Removed reference to EPA 6010B. The laboratory is discontinuing this test.
- Removed remaining references to SM2130B. This method is not performed by the laboratory.
- Added references to Hardness by SM2340B, since this Work Instruction is included as an Attachment. Title Page and Section 2.0. Revised existing reference to SM2340B to reflect current Standard Methods nomenclature (i.e., includes approval date of 1997 and editorial revision date of 2011). Section 15
- Included reference to Safety Data Sheets/SDSs. Section 5.2.
- Updated Lab Supplies section. Added nitrogen gas. Removed disposable graduated cylinders and detergent. Section 6.2.
- Removed references to Thermo trace instrument, and associated ISTD and instrument-specific configurations (e.g., automatic profile). This instrument has been removed from service. Section 6.1, Section 9.2.3, and Section 9.2.6.
- Added requirement to perform a torch alignment sequence following instrument warm-up, and removed instructions to run the "Automatic Profile" program. Sections 10.3.1 and 10.3.5 and Attachment 1
- Revised ICV %RSD criteria for EPA 200.7 from  $\leq 5\%$  to  $\leq 3\%$ . Section 9.2.3 and Attachment 3.
- Revised section on %RSD criteria for replicate exposures. Criteria changed to reflect method requirements (i.e., revised from 5% to reflect criteria of 30% - only for samples with detections above the reporting limit). This change stems from a 2013 Environmental Standards Audit finding. Section 9.2.11.
- Removed requirement to document IDOC on IDOC Form. Section 12.7.
- Updated standard tables in Attachment 5 and Attachment 6 and SOP text to reflect vendors, mixes, concentrations, and recipes currently in use.
- Removed remaining reference to MARRS software. This software is no longer used by the laboratory. Added information on current process using TALS. Section 6.1
- Added note that investigation is required if Total vs. Dissolved relationship is not met. Section 11.1.3
- Clarified section on physical interferences. Section 4.1.5
- Revised CCV criteria to reflect actual laboratory practice (which encompasses DOD %RSD criteria.) Added to Method Modification Section. Section 9.2.5 and Section 16.0
- Corrected IDL frequency in text of SOP to match current laboratory practice and Attachment 3 (i.e., changed from annually to quarterly). Added to Method Modification Section. Section 12.4 and Section 16.0
- Revised Preventative Maintenance Schedule to reflect actual laboratory practice. Attachment 4
- Clarified sections on performing dissolved metals. Added requirement that dissolved metals samples must be filtered in the field (prior to preservation). If the laboratory is requested to filter the samples, then appropriate narration must be added to the Narrative. Included requirement that samples must be allowed to sit for 24 hours after filtration/preservation. Added to Method Modification Section.

This change stems from a 2013 Environmental Standards Audit. Section 8.1 and Section 16

- Added information of laboratory's current practice of filtering all soil digestates and water digestates that have particulates, prior to analysis. Added requirement to document this filtration using the Batch Data Types in TALS. Added requirement to filter associated method blank and LCS/LCSD. This change stems from a 2013 Environmental Standards Audit. Section 10.3
- Added information on linear range studies. Added requirement to analyze a linear range standard if the instrument is not calibrated over the entire linear range. Section 9.2.1.1
- Added requirement to perform a dilution if a sample concentration is above the calibration range (or 90% of the linear range for a single point curve). Section 11.1.1


## DISSOLVED GASES IN WATER

(Methods: RSK-175 Modified)

### Approvals (Signature/Date):

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## 1.0 **Scope and Application**

This SOP gives the procedures for the determination of dissolved gases in water samples by gas chromatography with flame ionization detection (GC/FID) and thermal conductivity detection (GC/TCD).

A complete target analyte list, the reporting limits (RL), the method detection limits (MDL), and the accuracy and precision criteria associated with this procedure are provided in the TALS Method Limit Groups (MLGs).

This SOP was written by and for TestAmerica's Savannah laboratory.

## 2.0 **Summary of Method**

A 5mL volume of nitrogen is injected into a filled autosampler vial (22mL with Teflon-lined cap) while the same volume of liquid is withdrawn. The vial is then placed into the Tekmar 7000 headspace analyzer. A sample volume of 1mL of the headspace is transferred and injected into a capillary column connected to both a flame ionization detector (FID) and thermal conductivity detector (TCD). The FID is used as the primary detector for methane, ethane, and ethene and is more sensitive than the TCD. The TCD is used as the secondary detector for methane only, when the concentration of methane is above the calibration range of the FID. Standards are prepared and analyzed in the same manner as samples.

Note: Due to a TALS configuration requiring analytes to have discreet names, when methane is analyzed and reported from the TCD detector, it is referred to as "CH4-TCD".

This SOP is based on the following:

- EPA Region 1 SOP: RSKSOP-175, Rev. 2 (May 2004): *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique.*

## 3.0 **Definitions**

Refer to the Glossary Section of the *Quality Assurance Manual* (QAM) for a complete listing of applicable definitions and acronyms.

## 4.0 **Interferences**

### 4.1 **Procedural Interferences**

- 4.1.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus and can make identification and/or quantification of the target analytes difficult.
- 4.1.2 All sample collection containers are single-use disposable containers which limits the potential for contamination. All non-disposable labware must be scrupulously cleaned in



accordance with the posted Labware Cleaning Instructions (found in Attachment 6) to ensure it is free from contaminants and does not contribute artifacts.

4.1.3 High purity reagents and solvents are used to help minimize interference problems. Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

4.1.4 Instrument and/or method blanks are routinely used to demonstrate all reagents and apparatus are free from interferences under the conditions of the analysis.

#### 4.2 Matrix Interferences

4.2.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample matrix.

4.2.2 Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes. As such, samples known to be clean should be analyzed first. To prevent carryover into subsequent samples, analysis of reagent blanks may be needed after the analysis of a sample containing high concentrations of analytes.

4.2.3 The primary interferences will occur from analytes that are “degassed” from the sample into the headspace along with the target analytes. The non-target analytes will usually consist of gasoline constituents such as alkenes, branched alkanes, or BTEX compounds.

### 5.0 Safety

Employees must abide by the policies and procedures in the TestAmerica Environmental Health and Safety Manual (EHSM), the TestAmerica Savannah Addendum to the EHSM, and this document.

This procedure may involve hazardous materials, operations, and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to follow appropriate safety, waste disposal, and health practices under the assumption that all samples and reagents are potentially hazardous.

The analyst must protect himself/herself from exposure to the sample matrix. Many of the samples that are tested may contain hazardous chemical compounds or biological organisms. The analyst must, at a minimum, wear protective clothing (lab coat), eye protection (safety glasses or face shield), disposable gloves, and closed-toe, nonabsorbent shoes when handling samples.

#### 5.1 Specific Safety Concerns or Requirements

The toxicity or carcinogenicity of chemicals used in this method has not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized.

Hydrochloric acid is extremely hazardous as an oxidizer, a corrosive, a poison, and is reactive. Inhalation of the vapors can cause coughing, choking, irritation of the nose,

throat, and respiratory tract, breathing difficulties, and lead to pneumonia and pulmonary edema. Contact with the skin can cause severe burns, redness, pain, and deep ulcers. Vapors are irritating and can cause damage to the eyes. Contact with the eyes can cause permanent damage. Concentrated acids should only be used under a functioning fume hood.

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

All analysts that use compressed gases must be familiar with the MSDS/SDS for the gas that they are using, and they must have completed safety training on the use of systems under pressure.

## 5.2 Primary Materials Used

The following is a list of the materials used in this procedure, which have a serious or significant hazard rating, and a summary of the primary hazards listed in their MSDS/SDS.

**NOTE: This list does not include all materials used in the procedure.** A complete list of materials used in this procedure can be found in the Reagents and Standards Section and the Equipment and Supplies Section of this SOP

Employees must review the information in the MSDS/SDS for each material before using it for the first time or when there are major changes to the MSDS/SDS. Electronic copies of MSDS/SDS can be found using the “MSDS” link on the Oasis homepage, on the EH&S webpage on Oasis, and on the QA Navigator.

Material	Hazards	Exposure Limit <sup>1</sup>	Signs and Symptoms of Exposure
Hydrochloric Acid <sup>2</sup>	Corrosive Poison	5ppm Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
<sup>1</sup> Exposure limit refers to the OSHA regulatory exposure limit.			
<sup>2</sup> Always add acid to water to prevent violent reactions.			

## 6.0 Equipment and Supplies

### 6.1 Equipment and Instrumentation

Teledyne/Tekmar Headspace Autosampler – 7000HT

Gas Chromatograph – Agilent 6890, or equivalent (equipped with FID and TCD)

Columns:

FID – Restek RTU Plot 30m x 0.32mm id

TCD – Supelco Carboxen – PL 30m x 0.32 mm id

## 6.2 Analytical Data System / Software / Hardware

Chemstation software is used on a Windows-based PC to schedule and acquire data. CHROM software is used on a Windows-based PC to store, reduce/evaluate, and output the data to the laboratory's LIMS system (i.e., TALS). CHROM software has the capability of processing stored GC data by recognizing a GC peak within any given retention time window and comparing the retention time of the sample to the retention times of the standards analyzed under the same conditions. The software also allows calculation integration of the peak responses, response factors, construction of a linear regression calibration curve, calculation of response factor statistics (mean and standard deviation), and calculation of concentrations of analytes using either the calibration curve or the response factors.

## 6.3 Volumetric Labware

All volumetric labware must be verified in accordance with SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*. Refer to the table below for detailed descriptions of containers including type, use and verification criteria and frequency requirements. Refer to Attachment 12 for labware cleaning procedures.

<b>Volumetric Labware</b>	<b>Volume</b>	<b>Type (Quantitative / Qualitative)</b>	<b>Use</b>	<b>Verification Frequency</b>	<b>Verification Criteria</b>
Tedlar Bags	1L	Qualitative	Nitrogen Container	None	None
Autosampler Vials	22mL	QUANTITATIVE	Instrument Loading; Standard Preparation	Per Lot	Accuracy = 2% Precision = 1%
VOA Vials	43mL	Qualitative	Sample Collection	None	None
Gas Tight Syringes	Various	QUANTITATIVE	Reagent Measurement and Delivery	None (Received w/ COA)	None (Received w/ COA)
Volumetric Flasks (Class A)	Various	QUANTITATIVE	Dilution Preparation	None	None (Purchased Class A)

## 6.4 Sample Collection Containers

All sample collection containers are single-use disposable containers which limits the potential for contamination.

The routine sample collection containers supplied by the laboratory are:

40mL VOA vials with Teflon lined caps – purchased with Certificate of Analysis attesting to purity.

## 7.0 **Reagents and Standards**

### 7.1 **Expiration Dates**

Expiration dates (time from initial use or receipt to final use) for standard and reagent materials must be set according to the guidance in this SOP. Note: These are maximum expiration dates and are not to be considered an absolute guarantee of standard or reagent quality. Sound judgment must be used when deciding whether to use a standard or reagent. If there is doubt about the quality of a standard or reagent material, a new material must be obtained or the standard or reagent material verified. Data quality must not be compromised to extend a standard's life.

The expiration date of any standard or reagent must not exceed the expiration date of the standard or reagent that was used to prepare it.

### 7.2 **Reagents**

Reagents must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*.

Hydrochloric acid must be verified prior to use in accordance with the TestAmerica Solvent Lot Testing Program.

#### 7.2.1 Purchased Reagents

Laboratory Reagent Water – ASTM Type I; purged with nitrogen for 8 hours.

Nitrogen – Zero grade or better

Expiration:

Unopened: Manufacturer's expiration date

Opened: Manufacturer's expiration date

### 7.3 **Standards**

Standards must be prepared and documented in accordance with SOP SA-AN-041: *Reagent and Standard Materials Procedures*. Certificates of analysis or purity must be received with all purchased standards, and scanned and attached in TALS.

#### 7.3.1 Purchased Standards

The standards used for this procedure are comprised of Scotty II cylinders containing mixtures of various analytes in nitrogen or helium. The standards are diluted in nitrogen and injected into vials containing reagent water to prepare the calibration and QC samples.

The Scotty II cylinders used are:

Supelco Catalogue No.	Components	Concentration	Source
23470u	Methane, Ethane, Ethene	15ppmV in N <sub>2</sub>	Primary
23462	Methane, Ethane, Ethene	1% v/v in N <sub>2</sub>	Primary
501697	Methane	4% in helium	Primary
22562	Methane	99%	Primary

Storage: Room temperature

Expiration:

Unopened: Manufacturer's expiration date

Opened: Manufacturer's expiration date

### 7.3.2 Prepared Standards

The preparation of dissolved gas standards is based on the following equation:

$$ppmV = ug/L \times \frac{24.47}{MW}$$

Where:

ppmV = parts per million concentration on a volume per volume basis

ug/L = micrograms of analyte per unit volume

24.47 = molal gas constant @ STP

MW = molecular weight of analyte

A rearrangement of the equation gives the following:

$$ug/L = ppmV \times \frac{MW}{24.47}$$

To determine the mass (µg) of a standard where the ppmV concentration is known, insert the numbers and solve for µg/L. For example, a standard that has methane at 1% (10000ppmV) on a volume basis is converted to mass of methane per unit volume as follows:

$$ug/L = 10000ppmV \times \frac{16}{24.47} = 6539ug/L = 6539ng/mL$$

When preparing working standards, a total volume of 5.0mL of the prepared gas standard is always added to the autosampler vial at the same time that 5.0mL of reagent water is removed from the vial. Therefore, if a 22mL VOA vial is used, 5.0mL of the prepared gas standard is added to the full VOA vial while 5mL of reagent water is drawn off. That is, 5.0mL of prepared gas standard is added to essentially 17mL of reagent water.

The micrograms of methane per liter of water are determined as follows:

$$\frac{6539ng/mL \times 5.0mL}{17mL} = 1923ng/mL = 1923ug/L$$

#### 7.3.2.1 GC/FID Standards

Initial Calibration – Fill ten 22mL vials with reagent water. No headspace is allowed to be present in the vial.

Prepare the calibration standards using the amounts listed in the table, below. A 5mL syringe is used to collect the volume of standard in the Std Amount column. An additional volume of nitrogen is then added to this syringe to achieve a 5mL final volume. Invert the 22mL vial and puncture the Teflon cap with the gas-filled 5mL syringe. Puncture the cap with a second 5mL gastight syringe. Fill the 22mL sample vial with the standard while withdrawing the displaced water with the second syringe.

Calibration Levels	Std Amount (15ppmV)	Amount N <sub>2</sub> (mL)	Methane (ug/L)	Ethene (ug/L)	Ethane (ug/L)
Level 1	1.000mL	4.0	0.576	1.012	1.082
Level 2	2.500mL	2.5	1.441	2.529	2.705
Level 3	5.000mL	0.0	2.882	5.059	5.410
	<b>Std Amount (1%)</b>				
Level 4	0.100mL	4.9	38.46	67.31	72.12
Level 5	0.200mL	4.8	76.43	134.6	144.2
Level 6	0.400mL	4.6	153.8	269.2	288.5
Level 7	0.800mL	4.2	307.7	538.5	576.9
Level 8	1.000mL	4.0	384.6	673.1	721.0

Storage: N/A (single use)

Expiration: N/A (single use)

Note: Instrument may be calibrated higher based on the sensitivity of the detector.

#### 7.3.2.2 GC/TCD Standards

Initial Calibration – Fill seven 22mL vials with reagent water. No headspace is allowed to be present in the vial.

Prepare the calibration standards using the amounts listed in the table, below. A 5mL syringe is used to collect the volume of standard in the Std Amount column. An additional volume of nitrogen is then added to this syringe to achieve a 5mL final volume. Invert the 22mL vial and puncture the Teflon cap with the gas-filled 5mL syringe. Puncture the cap with a second 5mL gastight syringe. Fill the 22mL sample vial with the standard while withdrawing the displaced water with the second syringe.

Calibration Levels	Std Amount (1%)	Amount N <sub>2</sub> (mL)	CH <sub>4</sub> -TCD (ug/L)
Level 1	1.00mL	4.0	384.6
Level 2	2.00mL	3.0	769.2
Level 3	5.00mL	0.0	1923
	<b>Std Amount (4%)</b>		
Level 4	2.00mL	3.0	3077
Level 5	5.00mL	0.0	7692
	<b>Std Amount (99%)</b>		
Level 6	0.500mL	4.5	19039
Level 7	1.00mL	4.0	38078

Storage: N/A (single use)

Expiration: N/A (single use)

Note: The working range of the TCD can be extended by analyzing higher concentrations of standards. This is particularly important for samples with high levels of analytes. The error due to dilutions can be significant. Do not dilute the samples if the levels exceed the calibration by more than a factor of ten; instead extend the working range of the calibration.

## 8.0 **Sample Collection, Preservation, Shipment, and Storage**

### 8.1 **Aqueous Samples**

Aqueous samples are routinely collected with zero headspace in 40mL VOA vials containing 1:1 HCl preservative, sufficient to achieve pH < 2.

Samples must be iced at the time of collection and maintained at 0-6°C (less than 6°C but not frozen) until the time of analysis. Samples must be analyzed within as soon as possible upon collection, with a maximum holding time of 14 days.

NCMs must be initiated for samples collected in improper containers and containing improper or insufficient preservatives and/or de-chlorination agents. NCMs must be initiated for samples that are received containing headspace.

#### 8.1.1 Headspace Verification

At the time of receipt, each sample vial must be evaluated for headspace by holding the sample vial upside down and examining it for "bubbles".

- If no bubbles are present or the bubbles are less than 3mm in diameter, the vial is acceptable.



- If bubbles are present and greater than 3mm in diameter, initiate a Nonconformance Memo.

#### 8.1.2 pH Verification

For each sample, sacrifice one vial to determine the pH. The criterion for samples preserved with acid is pH<2. If the sacrifice vial is acceptable, the remaining vials are assumed to be acceptable; if the sacrifice vial is not acceptable, the remaining vials are deemed unacceptable also.

For each sample,

- Note the color change of the pH paper.
- If the pH is less than 2, the sample is acceptable. Record this information on the benchsheet.
- If the pH is greater than or equal to 2, the sample has not been properly preserved. Record this information on the benchsheet and initiate an NCM.

### 9.0 **Quality Control**

SOP SA-QA-017: *Evaluation of Batch QC Data* and the SOP Summary in Attachment 3 provide requirements for evaluating QC data.

#### 9.1 **Batch QC**

An analytical batch consists of up to 20 environmental samples and the associated QC items analyzed together within a 24 hour period.

The minimum QC items required for each extraction batch are: a method blank and a laboratory control sample (LCS), and a laboratory control sample duplicate (LCSD).

Due to the nature of this analysis, which requires consumption of the entire sample and varying concentration ranges per detector, matrix spikes (MS) and matrix spike duplicates (MSD) are not routinely performed unless specifically requested by the client. An LCSD is performed to provide batch precision data. MS/MSD are not performed for the TCD detector unless there is a site history indicating the samples are at a high enough concentration such that the TCD detector will be required for all samples in the batch.

Note: If an LCS and LCSD are performed, both QC items must be evaluated and reported. Acceptable recoveries (as well as %RPD) for both LCS and LCSD are required.

The routine container supplied for this method is a 40mL container. The entire vial is needed for analysis.

Batch QC must meet the criteria given in Attachment 3 of this SOP.

#### 9.2 **Instrument QC**

##### 9.2.1 Initial Calibration (ICAL)

The instrument must be calibrated in accordance with SOP SA-QA-016: *Evaluation of Calibration Curves*. This SOP provides requirements for establishing the calibration curve and gives the applicable formulas.

Instrument calibration is performed by analyzing a series of known standards. The calibration curve must consist of a minimum of 5 standards. The lowest level calibration standard must be at or below the reporting limit, and the remaining standards will define the working range of the analytical system.

Note: A minimum of 6 points is required for a quadratic curve. Higher order curves are not permitted.

The initial calibration standard concentrations currently in use in the laboratory are given in Section 7.3.2. Refer to Section 7.3.2 for the standard preparation instructions. Other standard concentrations may be used provided they support the reporting limit and are fully documented in accordance with SOP SA-AN-041.

#### 9.2.2.1 ICAL Criteria

The preferred method of quantitation is the average response factor. The relative standard deviation (%RSD) of the calibration standards must be  $\leq 25\%$  to be acceptable.

If one or more compounds do not meet the %RSD criterion, the next option is to evaluate a regression curve. If the regression curve option is chosen, the regression coefficient ( $r^2$ ) must be greater than or equal to 0.990 to be acceptable.

If these criteria are not met, then re-calibration is required before sample analysis can proceed.

#### 9.2.2 Second Source Initial Calibration Verification (ICV)

The calibration curve must be verified after the initial calibration is established, prior to any sample analyses, in accordance with SOP SA-QA-016 with a standard obtained from a different lot.

The initial calibration verification standard concentration currently in use in the laboratory is equivalent to Level 6 of the ICAL for the FID and Level 3 of the ICAL for the TCD. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

The ICV must be  $\leq 25\%D$  to be acceptable.

#### 9.2.3 Continuing Calibration Verification

The initial calibration curve must be verified at the beginning of each analysis batch, after every 24 hours or 20 samples – whichever is greater, and at the end of the analysis batch with a mid-level standard.

The CCV must be  $\leq 15\%D$  to be acceptable.

The continuing calibration verification standard concentration currently in use in the laboratory is equivalent to level 6 of the ICAL for the FID and 3 of the ICAL for the TCD. Refer to Section 7.3.2 for the standard preparation instructions. Another standard concentration may be used provided it is mid-level and fully documented in accordance with SOP SA-AN-041.

### 9.3 Corrective Action for Out-of-Control Data

When the quality control parameters do not meet the criteria set forth in this SOP, corrective action must be taken in accordance with SOP SA-QA-005: *Preventive and Corrective Action Procedures* and the QC Summary Table in Attachment 3. SOP SA-QA-005 provides contingencies for out-of-control data and gives guidance for exceptionally permitting departures from approved policies and procedures. Nonconformance Memos must be initiated to document all instances where QC criteria are not met and all departures from approved policies and procedures.

## 10.0 Procedure

### 10.1 Sample Preparation

Remove the samples from the refrigerator.

Fill a Tedlar bag with nitrogen. This will be the reservoir of nitrogen to be added to the vials.

Fill a 5.0mL gastight syringe with nitrogen from the Tedlar bag.

Quickly transfer the sample to a 22mL vial. Invert the 22mL vial and puncture the Teflon cap with the nitrogen-filled syringe. Puncture the cap with a second 5.0mL gastight syringe. Fill the 22mL sample vial with the nitrogen while withdrawing the displaced water with the second syringe.

Place each sample, calibration standard, and QC sample on the Tekmar 7000 Headspace autosampler.

### 10.2 QC Sample Preparation

#### 10.2.1 GC/FID QC Sample Preparation

10.2.1.1 Method Blank – Fill a 22mL vial with reagent water for the method blank. Prepare as a sample in accordance with Section 10.1.

10.2.1.2 Laboratory Control Standard (LCS) – Fill a 22mL vial with reagent water for the LCS. The standard used to prepare the LCS is made from Supelco 1%v/v in N<sub>2</sub> containing methane, ethane, and ethane. (Note: This mix also contains acetylene, carbon dioxide, and carbon monoxide; however, these analytes are not analyzed/reported using this procedure).

The LCS is prepared using the same technique utilized for the initial calibration, Section 7.3.2.1.1. Add 0.4mL of the Supelco 1%v/v in N<sub>2</sub> and 4.6mL of N<sub>2</sub> to the vial

containing the reagent water. Label the vial appropriately. Prepare as a sample in accordance with Section 10.1.

The theoretical concentrations are:

Analyte	Concentration (ug/L)
Methane	150
Ethane	270
Ethene	290

- 10.2.1.3 Matrix Spike(s) (MS/MSD) – Fill a 22mL vial with the sample chosen as the MS/MSD. The standard used to prepare the MS/MSD is made from Supelco Mix 216 containing methane, ethane, and thane. (Note: This mix also contains acetylene, carbon dioxide, and carbon monoxide; however, these analytes are not analyzed/reported using this procedure).

The MS/MSD is prepared using the same technique utilized for the initial calibration, Section 7.3.2.1.1. Add 0.4mL of the Supelco Mix 216 1%v/v in N<sub>2</sub> and 4.6mL of N<sub>2</sub> to the vial containing the samples designated as the MS and MSD. Label the vial appropriately. Prepare as a sample in accordance with Section 10.1.

The theoretical concentrations are:

Analyte	Concentration (ug/L)
Methane	150
Ethane	270
Ethene	290

## 10.2.2 GC/TCD QC Sample Preparation

- 10.2.2.1 Method Blank – Fill a 22mL vial with reagent water for the method blank. Prepare as a sample in accordance with Section 10.1. (Note: The same method blank is used for both the FID and TCD.)
- 10.2.2.2 Laboratory Control Standard (LCS) – Fill a 22mL vial with reagent water for the LCS. The standard used to prepare the LCS is made from Supelco Mix 216 containing methane. (Note: This mix also contains acetylene, carbon dioxide, carbon monoxide, and nitrogen; however, these analytes are not analyzed/reported using this procedure).

Note: The LCS is prepared from a different lot of standard than those used to prepare the calibration standards; therefore, it can be used as the initial calibration verification (ICV).

The LCS is prepared using the same technique utilized for the initial calibration, Section 7.3.2.1.1. Add 5.0mL of the Supelco Mix 216 to the vial containing the

reagent water. Label the vial appropriately. Prepare as a sample in accordance with Section 10.1.

The theoretical concentration is:

Analyte	Concentration (ug/L)
CH4-TCD	1923

- 10.2.2.3 Matrix Spikes (MS/MSD) – MS/MSD are not performed for the TCD detector unless there is a site history indicating the samples are at a high enough concentration such that the TCD detector will be required for all samples in the batch. Prepare as a sample in accordance with Section 10.1.

### 10.3 Analysis

#### 10.3.1 Instrument Operating Conditions

The analysis of dissolved gases in water is divided into separate analytical procedures requiring different columns, detectors, and GC conditions. The instrument conditions listed in this SOP are provided for guidance purposes. The actual conditions used by the laboratory may be slightly different from those listed here and must be documented in the instrument maintenance log, data system, and/or run log.

Instrument maintenance must be performed in accordance with Attachment 4 of this SOP.

The goal is to have maximum separation between the target compounds in the shortest run time while maintaining sufficient sensitivity to detect the target compounds at the reporting limit and MDL (if required).

#### Default Instrument Configuration

Methane, Ethane, and Ethene by GC/FID	
Parameter	Conditions
Column/Flow	RT-U-Plot 30m x 0.32mm id
Detector/Temperature	250°C
Injector Port Temperature	250°C
GC Temperature Program	40°C for 0 minutes 8°C per minute to 88°C Hold 0 minutes
Injection Volume	1.0mL

Methane by GC/TCD	
Parameter	Conditions
Column/Flow	Carboxen 1010 Plot 30m x 0.32mm id

Detector/Temperature	250°C
Injector Port Temperature	250°C
GC Temperature Program	40°C for 0 minutes 8°C per minute to 88°C Hold 0 minutes
Injection Volume	1.0mL

#### 10.3.1.1 Determination of Retention Time Windows

The procedure for the determination of retention time windows is given in SOP SA-QA-008: *Evaluation of Chromatographic Data*. Retention time windows (RTW), i.e., the length of time the instrument will scan for the analyte, must be established initially upon instrument set-up and verified quarterly.

Retention times (RT), i.e., the elution time of the analyte, are verified daily with the analysis of the ICAL or CCV. The retention time for the CCV must fall within the daily retention time window as defined in SOP SA-QA-008.

#### 10.3.2 Initial and Continuing Calibration

Calibrate the instrument using the standards and criteria described given in Section 9.2.1. Once the calibration has been established and verified with an ICV in accordance with Section 9.2.2, sample analysis may proceed.

Verify the calibration curve with a continuing calibration verification using the standards and criteria described given in Section 9.2.4.

#### 10.3.3 Sample Analysis

The sample extract must be injected using the same injection volume used for the calibration standards. Samples that are known to be relatively clean should be analyzed first. Samples suspected of containing high concentrations should be analyzed last. Instrument blanks may be analyzed after suspected high concentration samples to allow the detector response to stabilize.

The default procedure is to include QC items (method blank, LCS/LCSD, and MS/MSD) in determining the maximum number of samples in the clock.

#### 10.3.4 Example Analytical Sequence

An example analytical sequence is provided in Attachment 1.

### 11.0 Calculations / Data Reduction

#### 11.1 Data Reduction

Data evaluation must be performed in accordance with SA-QA-008: *Evaluation of Chromatographic Data*. This SOP includes specific information regarding the evaluation of chromatographic data, including the requirements for performing manual integrations and the evaluation of retention times.

Data must be evaluated in accordance with SOP SA-QA-002: *Data Generation and Review*.

#### 11.1.1 Target Analyte Identification

The judgment and experience of the analyst and his/her colleagues are important factors in the evaluation of chromatographic data. Inspect each chromatogram to ensure that the peaks are properly identified and that the correct areas have been associated with the corresponding standard peak RT in the data system tabulation.

The evaluation of chromatograms for target compounds must take into account the calibration of the analytical system (initial and continuing calibration response and retention times); the recovery and retention time shift of the surrogate compounds, whether the peak response falls within the working range of the calibration; and the integration of the peaks. The analyst must also take into account the results from the method blank and lab control sample before reporting quantitative data. SOP SA-QA-008: *Evaluation of Chromatographic Data* provides additional guidance for the evaluation of chromatographic data. This guidance is summarized in the following sections.

#### 11.1.2 Manual Integrations

Manual integrations must be documented in accordance with SOP SA-QA-008. Data systems should be adjusted to minimize operator intervention. All chromatographic peaks must be evaluated for overall peak shape and “reasonableness” of integration. Under no circumstances should manual integrations be used to change reasonable data system integrations in order to meet calibration or QC criteria.

#### 11.1.3 Dilutions

If the concentration of the methane exceeds the working range of the FID (defined by the highest standard in the initial calibration), the results from the TCD must be evaluated. If the concentration of ethane or ethene exceeds the working range of the FID, or if the concentration of methane exceeds the response of the TCD, then the sample must be diluted and reanalyzed. A dilution should bring the area of the largest peak of interest into the upper half of the calibration curve.

Do not inject more than one aliquot from a single vial. Sample dilutions are performed prior to transfer to the 22mL vial.

Unless otherwise specified by a client QAPP, results from a single analysis are reported as long as the largest target analyte (when multiple analytes are present) is in the upper half of the calibration range. When reporting results from dilutions, appropriate data flags must be used or qualification in a case narrative provided to the client.

For clients who require we provide lower detection limits, a general guide would be to report the dilution detailed above and one additional run at a dilution factor 1/10 of the



dilution with the highest target in the upper half of the calibration curve. For example, if samples analyzed at a 1/50 dilution resulted in a target in the upper half of the calibration curve, the sample would be analyzed at a dilution factor of 1/5 to provide lower reporting limits.

#### 11.1.3.1 Preparation of Dilutions

Prepare dilutions as follows:

- Remove the sample from the refrigerator. Note: Do not use a vial that has already been used or contains headspace.
- Partially fill a 50mL volumetric flask with reagent water.
- Using a gastight syringe, remove the volume of sample needed to prepare the dilution.

Dilution	Volume of Sample to Add (mL)
10	5
5	10
2	25

11.1.4 Using reagent water, bring the 50mL volumetric flask up to volume and gently pour the sample just to the top of the 22mL vial prepared for the dilution.

#### 11.1.5 Historical Data

Many of the laboratory's clients submit samples for repeat monitoring purposes. Prior to analysis, verify TALS Worksheet Notes and/or use the Historical Data Tracker Feature to determine if historical data is available for review.

### 11.2 Calculations

11.2.1 The calculations associated with batch QC determinations are given in SOP SA-QA-017. Applicable calculations include accuracy (% recovery) and precision (%RPD).

11.2.2 The calculations associated with initial and continuing calibrations and are given in SOP SA-QA-016. Applicable calculations include determination for: calibration factor, standard deviation, relative standard deviation, relative response factor, and relative standard deviation.

11.2.3 The concentration of the standard (ug analyte per liter of water) is plotted against the response (area) to prepare a calibration curve for each target analyte. The concentration of the sample is calculated using the following equation:

$$\text{ug/L(water)} = \text{ug/L(curve)} \times \frac{1.0\text{mL}}{V_i} \times \frac{22\text{mL}}{V_s} \times DF$$

Where:

ug/L(curve) = concentration from the curve

$V_i$  = volume of headspace injected

$V_s$  = volume of sample analyzed

DF = dilution factor

**Note: This calculation assumes all applicable unit correction factors are applied.**

## **12.0 Method Performance**

### **12.1 Reporting Limit Verification (RLV)**

At a minimum, RLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, RLVs must also be performed quarterly thereafter. For all other analytes and methods, RLVs must also be performed annually thereafter. Exceptions may be made for project-specific non-routine analytes.

### **12.2 Method Detection Limit (MDL) Study**

The MDL is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix and may not be achievable in all environmental matrices. The current MDLs associated with this procedure are given in the Method Limit Group (MLG) in TALS.

At a minimum, MDL Studies must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

Note: MDL Studies are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

### **12.3 Method Detection Limit Verification (MDLV)**

At a minimum, MDLVs must be performed initially upon method set-up in accordance with SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*.

For analytes and methods certified by DOD ELAP, MDLVs must also be performed quarterly thereafter. For all other analytes and methods, MDLVs must also be performed annually thereafter.

Note: MDLVs are not required for non-routine analytes provided results are not reported below the RL (i.e., MDL equals RL in TALS).

### **12.4 QC Limit Generation, Control Charting, and Trend Analysis**

The control limits for the batch QC items (LCS and MS/MSD) for this procedure are specified in the reference method and cannot be broadened; therefore, the laboratory defaults to the method-defined limits and does not utilize in-house or laboratory-derived limits for the evaluation of batch QC items.

Although the laboratory must default to the method-defined QC limits, control charting is a useful tool and is performed to assess analyte recoveries over time to evaluate trends. Control charting must be performed periodically (at a minimum annually) in accordance with SOP SA-QA-017: *Evaluation of Batch QC Data*.

#### 12.5 Demonstrations of Capability

Initial and continuing demonstration of capability must be performed in accordance with SOP SA-QA-006: *Training Procedures*.

Prior to performing this procedure unsupervised, each new analyst who performs this analysis must demonstrate proficiency per method/analyte combination by successful completion of an initial demonstration of capability. The IDOC is performed by the analysis of 4 consecutive LCSs that meet the method criteria for accuracy and precision. The IDOC must be documented and routed to the QA Department for filing.

Annual continuing demonstrations of capability (CDOCs) are also required per analyst per method/analyte combination. The CDOC requirement may be met by the consecutive analysis of four LCS all in the same batch, by the analysis of four LCS analyzed in four consecutive batches (in different batches on different days), via acceptable results on a PT study, or analysis of client samples with statistically indistinguishable results when compared to another certified analyst. The CDOC must be documented and routed to the QA Department for filing.

#### 12.6 Training Requirements

All training must be performed and documented in accordance with SOP SA-QA-006: *Training Procedures*.

Note: The SOPs listed in the Reference/Cross-Reference Section are applicable to this procedure. All employees performing this procedure must also be trained on these SOPs, and/or have a general understanding of these procedures, as applicable.

### 13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (e.g., examining recycling options, ordering chemicals based on quantity needed, preparing reagents based on anticipated usage and reagent stability, etc.). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual and the Savannah Addendum to the EHSM.

This procedure has been evaluated for opportunities to minimize the waste generated. Where reasonably feasible, pollution control procedures have been incorporated.

### 14.0 Waste Management

Waste management practices must be conducted consistent with all applicable federal, state, and local rules and regulations. All waste (i.e., excess reagents, samples, and method process wastes) must be disposed of in accordance with Section 9 of the

TestAmerica Savannah Addendum to the EHSM. Waste description rules and land disposal restrictions must be followed.

#### 14.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

- Excess aqueous samples – Dispose according to characterization on the sample disposal sheets. Neutralize non-hazardous samples before disposal into drain/sewer. Transfer hazardous samples (identified on disposal sheets) to the waste department for disposal.

#### 15.0 References / Cross-References

- SOP SA-AN-100: *Laboratory Support Equipment (Verification and Use)*
- SOP SA-AN-041: *Reagent and Standard Materials Traceability*
- SOP SA-QA-002: *Data Generation and Review*
- SOP SA-QA-005: *Preventive and Corrective Action Procedures*
- SOP SA-QA-006: *Training Procedures*
- SOP SA-QA-007: *Determination and Verification of Detection and Reporting Limits*
- SOP SA-QA-008: *Evaluation of Chromatographic Data*
- SOP SA-QA-016: *Evaluation of Calibration Curves*
- SOP SA-QA-017: *Evaluation of Batch QC Data*
- TestAmerica Savannah Quality Assurance Manual
- TestAmerica Environmental Health and Safety Manual (CW-E-M-001)
- TestAmerica Savannah Addendum to the Environmental Health and Safety Manual
- Paper: *Dissolved Oxygen and Methane in Water by a GC Headspace Equilibration Technique* (Kampbell and Wilson, USEPA; Vendegrift, NSI; March 1989)
- EPA Region 1 SOP: RSKSOP-175, Rev. 2 (May 2004): *Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique*.
- EPA Region 1 SOP: RSKSOP-194, Rev. 3 (January 2005): *Gas Analysis by Micro Gas Chromatographs (HP Series P200H and MTI P200 GC)*.

#### 16.0 Method Modifications and Clarifications

The laboratory performs a modified RSK-175 method and has incorporated several procedural and QC modifications into this SOP. Modifications include bottle types and preservatives, sample preparation procedures, instrument configuration, calibration criteria, CCV frequency and criteria, and batch QC frequency and criteria. Other modifications and/or clarifications are listed below.

- 16.1 The RSK-175 method includes the analysis of several gases. The laboratory only performs methane, ethane, and ethene by this procedure. Dissolved nitrogen is not measured by this procedure since nitrogen is used for the headspace gas. Oxygen is not determined because of the high probability of contamination from the atmosphere and the narrow range of concentrations that may be determined by the TCD.
- 16.2 The RSK-175 method specifies to use Henry's Law to calculate concentrations. Henry's

Law states the mass of a slightly soluble gas that dissolves in a definite mass of a liquid at a given temperature is very nearly directly proportional to the partial pressure of that gas. This law is applicable to dilute concentrations of gases at pressures of one atmosphere or less. The laboratory assumes that Henry's Law applies "equally" to the field samples and to the standards analyzed under essentially the same conditions (temperature, pressure, and headspace volume).

- 16.3 Due to the nature of this analysis, which requires consumption of the entire sample and varying concentration ranges per detector, matrix spikes (MS) and matrix spike duplicates (MSD) are not routinely performed unless specifically requested by the client. An LCSD is performed to provide batch precision data. MS/MSD are not performed for the TCD detector unless there is a site history indicating the samples are at a high enough concentration such that the TCD detector will be required for all samples in the batch.
- 16.4 Second detector confirmation is only available for high concentrations of methane. Identification and quantitation of ethane, ethene, and low concentrations of methane are performed using a single detector.
- 16.5 The laboratory has not identified a readily available ISO Guide 34 vendor or a second vendor for these standards to serve as the 2<sup>nd</sup> Source Initial Calibration Verification (ICV). The ICV is prepared from a second lot, as opposed to a second vendor

**17.0 Attachments**

The following Tables, Diagrams, and/or Validation Data are included as Attachments:

- Attachment 1: SOP Summary
- Attachment 2: Sample Collection, Preservation, and Holding Time Table
- Attachment 3: QC Summary
- Attachment 4: Instrument Maintenance and Troubleshooting
- Attachment 5: Example Calculations
- Attachment 6: Glassware Cleaning Procedures

## Attachment 1: SOP Summary

### Sample Preparation and Analysis Summary

A 5mL volume of nitrogen is injected into a filled autosampler vial (22mL with Teflon-lined cap) while the same volume of liquid is withdrawn. The vial is then placed into the Tekmar 7000 headspace analyzer. A sample volume of 1mL of the headspace is transferred and injected into a capillary column connected to both a flame ionization detector (FID) and thermal conductivity detector (TCD). The FID is used as the primary detector for methane, ethane, and ethene and is more sensitive than the TCD. The TCD is used as the secondary detector for methane only, when the concentration of methane is above the calibration range of the FID. Standards are prepared and analyzed in the same manner as samples.

Analyte	Molecular Weight	Detector
Methane and CH <sub>4</sub> -TCD	16	FID and TCD
Ethane	30	FID
Ethene	28	FID

### Example Analytical Sequence

Description	Comments
Initial Calibration	24-hour clock begins with injection of 1 <sup>st</sup> level of FID ICAL
ICV	Second Source
Samples & Batch QC Items	Up to 20 injections, not including QC. Not to exceed 24 hours.
CCV	Level 6 for the FID and 4 for the TCD 24-hour clock begins with injection of the first CCV
Samples & Batch QC Items	Up to 20 injections, not including QC. Not to exceed 24 hours.
CCV	Level 6 for the FID and 3 for the TCD 24-hour clock begins with injection of the first CCV
Samples & Batch QC Items	Up to 20 injections, not including QC. Not to exceed 24 hours.
CCV	Level 6 for the FID and 3 for the TCD 24-hour clock begins with injection of the first CCV

**Attachment 2:**  
**Sample Collection, Preservation, and Holding Time Table**

<b>Matrix</b>	<b>Routine Sample Container</b>	<b>Routine Sample Size</b>	<b>Minimum Sample Size</b>	<b>Dechlorination Agent</b>	<b>Chemical Preservation</b>	<b>Thermal Preservation</b>	<b>Holding Time</b>
Water	40mL VOA Vial	40mL	40mL	None	1:1 HCl, to pH <2	0-6°C <sup>1</sup>	14 days

<sup>1</sup>Samples are collected on ice and maintained at <6°C with no frozen samples.



**Attachment 3:  
QC Summary**

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL) Minimum 5 standards	Initially, and when ICV/CCV fails	$\%RSD \leq 25\%$ $r^2 \geq 0.990$	Recalibrate
2 <sup>nd</sup> Source Initial Calibration Verification (ICV)	After each ICAL	$\%D \leq 25\%$	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of each 24-hour clock, and after every 20 samples	$\%D \leq 15\%$	- Reprepate standards - Re-analyze affected samples - Recalibrate
Batch Definition	Processed together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	<RL	Refer to SOP SA-QA-017
Lab Control Sample (LCS)	One per batch	Within MLG Limits	Refer to SOP SA-QA-017
Laboratory Control Sample Duplicate (LCSD)	One per batch	Within MLG Limits	Refer to SOP SA-QA-017
Matrix Spike (MS)	1 per batch, if requested	Within MLG Limits	Refer to SOP SA-QA-017
Matrix Spike Duplicate (MSD)	1 per batch, if requested	Within MLG Limits	Refer to SOP SA-QA-017
Initial Demonstration of Capability (IDOC)	Initially; Per analyst / matrix / method / analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006  Note: Unsupervised work must not begin until acceptable IDOC is obtained.

QC Item	Frequency	Criteria	Corrective Action
Continuing Demonstration of Capability (CDOC)	Annually; Per analyst / matrix / method / analyte combination	Refer to SOP SA-QA-006	Refer to SOP SA-QA-006
Reporting Limit Verification (RLV)	Upon method/instrument set-up, per analyte/method/matrix combination.  Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Method Detection Limit Study (MDL Study)	Upon method/instrument set-up, per analyte/method/matrix combination  - MDL Study must be performed over a period of several days.	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
MDL Verification (MDLV)	Upon method/instrument set-up, per analyte/method/matrix combination. Then quarterly thereafter (for DOD ELAP) or annually thereafter (for NELAC)	Refer to SOP SA-QA-007	Refer to SOP SA-QA-007
Retention Time Window (RTW) Determination	Annually, after major instrument maintenance, and with each new column	Refer to SOP SA-QA-008	Refer to SOP SA-QA-008

## **Attachment 4: Instrument Maintenance and Troubleshooting**

### **Instrument Labeling**

Each instrument must be labeled with its name or ID (e.g., MSA, ICP-D, etc.). Additionally, non-operational instruments must be isolated from service or marked as being out of service. Each piece of equipment has an "Operational / Not Operational" sticker that is used for this purpose.

### **Maintenance Log**

A maintenance log must be established for each piece of equipment used in the laboratory.

All maintenance that is performed on the instrument must be recorded in the log including:

- analyst or technician performing the maintenance
- date the maintenance was performed
- detailed explanation of the reason for the maintenance
- resolution of the problem and return to control
- all service calls from instrument representatives

### **Preventive Maintenance**

Refer to the instrument manufacturer's guides for trouble-shooting items. There are no routine preventive maintenance items performed by the laboratory.

### **Troubleshooting**

Troubleshooting should be documented as outlined above. If possible, troubleshooting is best performed in a step-wise manner to systematically isolate instrument components. Refer to the instrument manufacturer's guides for specific information and strategies. Enlist assistance from technical and/or department management as needed.

### **Contingency Plan**

In the event of instrument failure, the analytical technique may be switched to an alternate approved technique (such as manual colorimetric determination as opposed to automated colorimetric determination), or samples may be shipped to another properly certified or approved TestAmerica location.

## Attachment 5: Example Calculations

The preparation of dissolved gas standards is based on the following equation:

$$\text{ppmV} = \text{ug/L} \times (24.47/\text{MW})$$

Where:

ppmV = parts per million concentration on a volume per volume basis

ug/L = micrograms of analyte per unit volume

24.47 = molal gas constant @ STP

MW = molecular weight of analyte

A rearrangement of the equation gives the following:

$$\text{ug/L} = \text{ppmV} \times (\text{MW}/24.47)$$

To determine the mass (ug) of standard where the ppmV concentration is known, insert the numbers and solve for ug/L. For example, a standard that has methane at 15ppmV on volume basis is converted to mass of methane per unit volume:

$$\text{ug/L} = 15\text{ppmV} \times (16/24.47) = 9.81\text{ug/L} = 9.81\text{ng/mL}$$

If 5.0mL of this gas is added to 17mL of water (the sample vial holds 22mL; we remove 5mL adding the standard), the micrograms of methane per liter of water is:

$$\frac{9.81\text{ng/mL} \times 5.0\text{mL}}{22\text{mL}} = 2.23\text{ng/mL} = 2.34\text{ug/L}$$

The above procedure is performed by varying the amount of gas standard added to each vial to generate a calibration curve. From the calibration curve the following average response factors were generated.

FID DETECTOR (ethene)

0.0000264

FID DETECTOR (ethane)

0.0000255

TCD DETECTOR (CH4-TCD)

0.0003534

Sample concentration in ug/L = Response Factor X Area X Dilution Factor

Insert the values from the chromatogram of sample 2952 (MW-7):

$$\text{Methane (ug/L)} = 0.0003534 \times 8695476 \times 1 = 3073 = 3100\text{ug/L}$$

$$\text{Ethene (ug/L)} = 0.0000264 \times 7455508 \times 1 = 196 = 196\text{ug/L}$$

$$\text{Ethane (ug/L)} = 0.000026 \times 146542 \times 1 = 3.81 = 3.81\text{ug/L}$$

## Attachment 6: Glassware Cleaning Procedures

### GLASSWARE CLEANING PROCEDURES

#### VOLATILES DEPARTMENT

1. Rinse glassware 3 times thoroughly with DI water.
2. Place glassware, top-down, within storage rack and allow to air dry.
3. If glassware was used to prepare waste sample, use FL-70 and water to scrub glassware and follow previous steps.

FVM008:05.14.14:2



## **18.0 Revision History**

### Summary of Changes:

- Minor editorial and formatting changes made.
  - o Updated SOP references to reflect current document control number designations.
  - o Replaced reference to LIMS with TALS.
  - o Clarified that methane reported from the TCD detector is referred to as CH<sub>4</sub>-TCD
- Added reference to Safety Data Sheets/SDS. Sections 5.1 and 5.2
- Added requirement to scan/attach standard COAs to TALS. Section 7.3
- Adjusted sample collection and storage conditions to reflect 0-6°C. Section 8.1 and Attachment 2
- Changed volume of routine container from 22mL to 40mL. Section 9.1
- Due to the volatile nature of the target analytes, removed requirement to allow samples to come to room temperature prior to preparation. Section 10.1
- Modified instructions on preparation of dilutions and specified that sample dilutions are performed prior to transfer to the 22mL vial. Section 11.1
- Include additional information detailing descriptions of volumetric containers including type, use and verification criteria and frequency requirements. Section 6.3
- Added Glassware Cleaning Procedures. Attachment 7
- Added note that readily available ISO Guide 34 vendor has not been identified. Section 16.5
- Removed reference to maintenance contracts, as these are no longer maintained by the laboratory. Removed reference to duplicate instrumentation. Attachment 4

**APPENDIX B**  
EXAMPLE DOCUMENTATION




**TestAmerica**  
THE LEADER IN ENVIRONMENTAL TESTING

<b>Client Information</b>		Sampler:		Lab PM: Atkins, Amy		Carrier Tracking No(s):		COC No: 640-47715-11114.1																			
Client Contact: Mr. James Roehrig		Phone:		E-Mail: amy.atkins@testamericainc.com				Page: Page 1 of 8																			
Company: GZA GeoEnvironmental, Inc.				<b>Analysis Requested</b>						Job #:																	
Address: 249 Vanderbilt Ave		Due Date Requested:		<div>Field Filtered Sample (Yes or No)</div> <div>Perform MS/MSD (Yes or No)</div> <div>8260C - Site VOHs</div> <div>5310C - Total Organic Carbon</div> <div>RSK_175 - MEE (Dissolved Gases)</div> <div>6010B - Dissolved Iron</div> <div>8260C - Site VOHs plus BTEXM</div> <div>Total Number of containers</div>						<b>Preservation Codes:</b> A - HCL B - NaOH C - Zn Acetate D - Nitric Acid E - NaHSO4 F - MeOH G - Amchlor H - Ascorbic Acid I - Ice J - DI Water K - EDTA L - EDA M - Hexane N - None O - AsNaO2 P - Na2O4S Q - Na2SO3 R - Na2S2SO3 S - H2SO4 T - TSP Dodecahydrate U - Acetone V - MCAA W - ph 4-5 Z - other (specify) <b>Other:</b>																	
City: Norwood		TAT Requested (days):																									
State, Zip: MA, 02062																											
Phone: 781-278-5734(Tel) 781-278-5701(Fax)		PO #: 3669																									
Email: james.roehrig@gza.com		WO #:																									
Project Name: HP-San German IB		Project #: 64004374																									
Site:		SSOW#:																									
<b>Sample Identification</b>		<b>Sample Date</b>		<b>Sample Time</b>		<b>Sample Type (C=Comp, G=grab)</b>		<b>Matrix (W=water, S=solid, O=waste/oil, BT=Tissue, A=Air)</b>		<b>Field Filtered Sample (Yes or No)</b>		<b>Perform MS/MSD (Yes or No)</b>		<b>8260C - Site VOHs</b>		<b>5310C - Total Organic Carbon</b>		<b>RSK_175 - MEE (Dissolved Gases)</b>		<b>6010B - Dissolved Iron</b>		<b>8260C - Site VOHs plus BTEXM</b>		<b>Total Number of containers</b>		<b>Special Instructions/Note:</b>	
						Preservation Code:																					

Field Audit Checklist  
HP Voluntary Remediation Project  
San German, PR

24065.15

 <p>GZA GeoEnvironmental, Inc. 249 Vanderbilt Ave Norwood, MA 02062 781-278-3700</p>				Project Number:		GZA: 24065.15				
				Project Name:		HP Voluntary Remediation Project				
				Location:		San German, PR				
				Field and Company Personnel:						
<b>FIELD EQUIPMENT/CALIBRATION DATE</b>										
In-Situ Smart Troll:				Water Level Indicators:						
Sensors: pH, ORP, Temperature, SC, & optical DO										
				Pumps:				<b>FIELD CONDITIONS</b>		
Calibrated								Date:		
								Weather/Conditions:		
<b>WELLS TO BE SAMPLED:</b>										
Well ID:										
Sampled?:										
<b>LABORATORY PARAMETERS TO BE ANALYZED:</b>										
Well ID:										
Complete <sup>1</sup> /Limited <sup>2</sup> ?:										
<b>FIELD PARAMETERS TO BE ANALYZED:</b>										
Well ID:										
Field <sup>3</sup> Analyzed?:										
<b>QUALITY CONTROL SAMPLES:</b>										
QC Samples:	Field Dup	MS/MSD	Trip Blanks:							
Well ID:			Field Blanks:							
Well ID:			Equipment Blanks:							
<b>NOTES (e.g., general observations, changes to SOPs and/or sampling programs, etc.):</b>										
Name:				Signature:				Date:		

1. Complete Lab Parameter Set includes: chlorinated volatile organic compounds (cVOCs), dissolved iron, sulfate, methane, ethene, ethane, total organic carbon (TOC), nitrate, and chloride.
2. Limited Lab Parameter Set includes: cVOCs and TOC.
3. Field Parameters include: dissolved oxygen (DO), oxidation-reduction potential (ORP), and pH.

## Shipping Order Form - Bottle Order



TestAmerica Tallahassee  
2846 Industrial Plaza Drive  
Tallahassee, FL 32301  
Phone (850) 878-3994 Fax (850) 878-9504

Shipping Order ID: 47715

Ship Via: FedEx International Economy

Due On: 10/22/2014 11:59:00PM

Due After: 10/20/2014 12:00:00AM

### Ship To Information

Project Manager: Amy Atkins  
Company Name: GZA GeoEnvironmental Inc  
Attention: Attn: Jessica Yeager - GUEST (HOLD FOR PICKUP)  
Address 1: 1150 Caribe Ave  
Address 2:  
Address 3:  
City: Ponce  
State: PR  
Zip: 00716  
Phone #: 781-278-5833  
Project Ref: HP-San German IB

### Notes to Bottle/Shipping Department

☐ Rebill Freight

☐ Labels on Coolers

Please notify us immediately if an error is found in shipment

CLIENT HAS LOTS OF EXTRA COOLERS -  
PLEASE PACK 'EM TIGHT - DON'T LEAVE  
ROOM FOR ICE

Pre-Label with Sample ID's and Pack in Sets

Label Bags and Coolers with Sample ID's

Include 3 Liters of DI Water

Send FEDEX International Economy: Hold  
for pickup at the Aguadilla, PR (BQN),  
FEDEX Service  
Center.

CALL PM WITH TRACKING NUMBER  
AFTER SHIPPING

Shipping Assets

Assets	Quantity	Description	Filled
			<input type="checkbox"/>

**Bottle Order Information**

Bottle Order: HP-San German IB - October Semiannual  
Bottle Order #: 11114  
Request From Client: 10/3/2014  
Date Order Posted: 9/25/2013 10:42:07AM  
Order Status: Shipped  
Prepared By: Matt Jones  
Deliver By Date: 10/22/2014 11:59:00PM  
Lab Project Number: 64004374

**Order Completion Information**

Creator: Matt Jones  
Filled by: Jeremy Gaskin  
Sent Date: 10/17/2014 8:39:38AM  
Sent Via: FedEx International Economy  
Tracking #: 531005800500

Sets	Bottles/Set	Qty	Bottle Type Description	Preservative	Method	Matrix	Sample Type	Comments	Lot #
35	3	105	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs	Water	Normal		
35	3	105	Voa Vial 40ml Amber - with Sulfuric Acid	Sulfuric Acid	5310C - Total Organic Carbon	Water	Normal		
35	1	35	Plastic 125mL - unpreserved	None	353.2 - Nitrate	Water	Normal		
35	1	35	Plastic 125mL - with Sulfuric Acid	Sulfuric Acid	353.2 - Nitrate	Water	Normal		
25	2	50	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	RSK_175 - MEE (Dissolved Gases)	Water	Normal		
25	3	75	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs	Water	Normal		
25	3	75	Voa Vial 40ml Amber - with Sulfuric Acid	Sulfuric Acid	5310C - Total Organic Carbon	Water	Normal		
25	1	25	Plastic 250ml - with Nitric Acid	Nitric Acid	6010B - Dissolved Iron	Water	Normal		
25	1	25	Plastic 250ml - unpreserved	None	300_ORGFM_28D - Chloride and Sulfate	Water	Normal		
25	1	25	Plastic 125mL - unpreserved	None	353.2 - Nitrate	Water	Normal		
25	1	25	Plastic 125mL - with Sulfuric Acid	Sulfuric Acid	353.2 - Nitrate	Water	Normal		
5	3	15	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs	Water	Field Blank		
5	3	15	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs	Water	Equipment Bla		
15	0	0	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs	Water	Trip Blank		
1	3	3	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs plus BTEXM	Water	Normal		
1	3	3	Voa Vial 40ml Amber - with Sulfuric Acid	Sulfuric Acid	5310C - Total Organic Carbon	Water	Normal		

Please notify us immediately if an error is found in shipment

1	1	1	1	Plastic 125mL - unpreserved	None	353.2 - Nitrate	Water	Normal	
1	1	1	1	Plastic 125mL - with Sulfuric Acid	Sulfuric Acid	353.2 - Nitrate	Water	Normal	
1	3	3	3	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	8260C - Site VOHs plus BTEXM	Water	Normal	
1	1	1	1	Plastic 250ml - with Nitric Acid	Nitric Acid	6010B - Dissolved Iron	Water	Normal	
1	3	3	3	Voa Vial 40ml Amber - with Sulfuric Acid	Sulfuric Acid	5310C - Total Organic Carbon	Water	Normal	
1	2	2	2	Voa Vial 40ml - Hydrochloric Acid	Hydrochloric Acid	RSK_175 - MEE (Dissolved Gases)	Water	Normal	
1	1	1	1	Plastic 250ml - unpreserved	None	300_ORGFM_28D - Chloride and Sulfate	Water	Normal	
1	1	1	1	Plastic 125mL - unpreserved	None	353.2 - Nitrate	Water	Normal	
1	1	1	1	Plastic 125mL - with Sulfuric Acid	Sulfuric Acid	353.2 - Nitrate	Water	Normal	

#### Notes to Field Staff:

#### Health and Safety Notes:

Preservative

Comment

Hydrochloric Acid

CAUTION! CONTAINS 1:1 HYDROCHLORIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

Nitric Acid

CAUTION! STRONG OXIDIZER! CONTAINS 1:1 NITRIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

Sulfuric Acid

CAUTION! CONTAINS 1:1 SULFURIC ACID. Avoid skin and eye contact. If contact is made, FLUSH IMMEDIATELY with water.

Relinquished By	Company	Date	Time	Received By	Company	Seal #:
Relinquished By	Company	Date	Time	Received By	Company	Seal #:

Please notify us immediately if an error is found in shipment

***Custody Seal***

DATE

SIGNATURE

**TestAmerica**  
THE LEADER IN ENVIRONMENTAL TESTING  
**751279**

**TestAmerica**  
THE LEADER IN ENVIRONMENTAL TESTING  
**751279**



**APPENDIX C**  
**GENERAL LABORATORY QUALITY CONTROL GUIDELINES**

The analytical laboratory selected to perform analyses for environmental samples collected at the Site must be capable of providing complete environmental analytical services consistent with USEPA protocols. The selected laboratory should implement QA/QC procedures consistent with the Region 2 SOPs, this generic QAAP, and specific analytical method SOPs.

All analytical data will be verified prior to being released by the Laboratory. Verification will include both editorial and technical reviews. A final review of the data package will be performed and the approved data package will be signed by the project manager, or designee, when complete.

The Laboratory shall communicate with GZA project manager, QA officer, or project chemist by telephone or via email as necessary throughout the process of sample scheduling, shipment, analysis and data reporting, to ensure that samples are properly processed. This shall include immediately notifying GZA of any irregularities with samples or sample paperwork received, noting discrepancies between paperwork and verbal orders placed, problems encountered in sample analyses that could affect data quality or schedule, and any laboratory conditions that may impact the timeliness of analyses or data reporting. The Laboratory shall notify GZA in advance regarding any data that could potentially be late and shall specify an estimated delivery date. In particular, the Laboratory should contact GZA project manager or QA officer before proceeding to the analysis if any of the situations occur:

- Relative standard deviation (%RSD) >90% or average relative response factor (RRF) < 0.05 during initial calibration for any project target analytes;
- Percent difference (%D) > 90% or RRF < 0.05 during initial and continuing calibration verification for any project target analytes;
- Internal standard (IS) area counts < 25% of 12-hr IS area, or if performance exhibits a major abrupt dropoff;
- Any surrogate, Laboratory Control Sample (LCS), or LCS Duplicate (LCSD) recovery < 10%;
- Broken sample bottles;
- Holding time is exceeded or preservation does not meet requirement; and
- Revision to the SOPs is made.

**APPENDIX D**  
**SAMPLING STANDARD OPERATING PROCEDURES (GZA)**

## **OVERBURDEN "MACHINE OPERATED HOLLOW-STEM AUGERING" BORING**

### **PURPOSE**

To establish standard operating procedures for the advancing of shallow earth borings employing the hollow-stem auger method for geotechnical explorations and hazardous waste site investigations.

### **EQUIPMENT AND MATERIALS**

The driller shall be capable of providing power-driven sectional hollow-stem auger flights with a minimum inside diameter of three and three-quarter inches (3-3/4"). In addition, the following equipment shall be present:

- Drill rods, minimum size equivalent to the "A" rod, (one- and five-eighths-inch [1-5/8"] O.D. and one and one-eighth inch [1-1/8"] I.D.)
- Hollow-stem auger plug
- Drive hammer one (1) three hundred pound (300 lb) and one (1) one hundred forty pound (140 lb) plus or minus five pounds ( $\pm 5$  lbs)
- Two-inch (2") O.D. split spoon sampler
- Roller bit and diamond corer bit
- Water tank and pump

### **PROCEDURES**

The boring is advanced by rotating a single section of hollow-stem auger into the soil to desired depth or the limit of the auger section. To continue advancing the borehole, additional auger flights are added one at a time and this sequence is repeated until the required depth is reached. When an obstruction is met, the driller may be required to attempt to penetrate the obstruction by the use of a roller bit or by coring. If attempts to penetrate obstruction are unsuccessful, boring will be abandoned.

Hollow-stem auger techniques should be employed without the use of drilling water while drilling on hazardous waste sites.

If water is deemed necessary by driller, its use must be approved by the drilling inspector. Drilling

water quality should be evaluated by collecting samples for appropriate analyses. When water is used to advance boring, it must not be recirculated back into the boring, unless specifically authorized by the drilling inspector.

The auger plug must be in place at the auger head while borehole is being advanced to prevent soil from being transported through auger.

## **RECORDS AND DOCUMENTATION**

The details of the boring shall be recorded on the GZA boring log (copy attached).

## **SPECIAL NOTES**

Whenever standard operating procedures are varied, it shall be recorded. The drilling inspector should also record any detected odor from boring, and depth encountered.

Hollow-stem auger borings often provide the simplest method of soil investigation and sampling. However, depths of auger investigations are limited by groundwater conditions, soil characteristics, and the equipment used.

## **APPLICABLE STANDARDS AND REFERENCES**

ASTM D1452-80 Soil Investigation and Sampling by Auger Borings

## **OVERBURDEN BORING: "WASH AND DRIVE BORINGS"**

### **PURPOSE**

To establish standard operating procedures for advancing earth borings employing the wash boring method and driven casing. Wash boring is a boring system by which material loosened by a bit is borne to the surface in the annular space between the bit and casing by water forced down through the pipe bearing the bit.

### **EQUIPMENT AND MATERIALS**

The driller shall be capable of providing a power driven rotary machine, equipped with five-foot (5') sections of drive casing, with a minimum I.D. of three inches (3"). In addition, the following equipment shall be present:

- Drill rods; minimum size drill rod shall be equivalent to the A rod (one- and five-eighths-inch (1-5/8") O.D. and one and one-eighth-inch [1-1/8"] I.D.)
- Roller bit and diamond corer bit
- Drive hammer one (1) three hundred pound (300 lb) and one (1) one hundred forty pound (140 lb) plus or minus 5 pounds ( $\pm 5$  lbs)
- Two-inch (2") O.D. split spoon sampler
- Water tank and pump

### **PROCEDURES**

To start a borehole, a five-foot (5') length of casing is driven into the soil with the three hundred pound (300 lb) hammer falling twenty-four inches (24") until its top end is at the proper elevation. Hammer blows required to advance the casing each twelve inch (12") increment should be recorded on the boring log. To cut the loose soil, the hollow rod bearing the bit is moved up and down through the drill rod and the soil in the casing is brought to the surface. Casing sections are added one at a time, and the above sequence is repeated until the required depth is reached. When an obstruction is met, the driller may be required to attempt to penetrate the obstruction by the use of a roller bit or by coring. If attempts to penetrate obstruction are unsuccessful, boring will be abandoned. The borehole may be advanced uncased in cohesive materials with the specific authorization of the drilling inspector. Stabilizing the borehole with drilling fluid other than water must also be approved by drilling inspector.

## **RECORDS AND DOCUMENTATION**

All details of the boring shall be recorded on the GZA boring log (copy attached).

## **SPECIAL NOTES**

Whenever standard operating procedures are varied, it shall be recorded. Any observed change in color, volume and/or viscosity of the returning drilling fluid is indicative of a soil change and should be recorded by the drilling inspector.

## **APPLICABLE STANDARDS AND REFERENCES**



## **ROCK CORE DRILLING**

### **PURPOSE**

To obtain bedrock core samples for geological classification and to provide a borehole of sufficient diameter for geohydrologic testing (pressure permeability, geophysical) of the bedrock and installation of monitoring devices.

### **EQUIPMENT AND MATERIALS**

- Appropriate hydraulic rotary drilling rig and drilling tools
- Double-tube, core barrel with appropriate diamond bit (NX, NQ size or as required by project)
- Appropriate wooden boxes specifically constructed to hold and store rock core

### **PROCEDURES**

1. Upon encountering refusal at the specified borehole locations, the casing shall be firmly seated on the rock and washed out before inserting the diamond bit core barrel.
2. The diamond bit core barrel shall be started in the hole and the rock shall be drilled in continuous five-foot length (5') intervals (runs) until the required depth is reached. The holes shall be carried into the bedrock to a depth sufficient to permit the engineer to determine to his satisfaction the character of the rock penetrated.

Drilling methods employed shall be adjusted continuously to obtain maximum core recovery. This may include adjustments to rate of flow of drilling fluid, rotation rate, and down pressure. Variations in bit types, in terms of diamond size, matrix and configuration of water ports may also be required to maximize core recovery.

3. When the core run is complete, the barrel shall be withdrawn from the hole and opened. The core shall be stored in an approved core box before the drilling is continued and labeled as described below. Cores shall be carefully handled to insure their proper identification and placement in the order in which they are removed from the hole. Care shall be taken to recover as large a percentage of unbroken core as possible.
4. The rock cores shall be placed in suitable wooden boxes so partitioned that individual core

runs and cores from each boring will be kept separate. The core is placed in the core box in book fashion with the top of the run at the upper left corner and the remaining core placed sequentially from left to right and from the rear (nearest the cover hinge) of the box to the front. The core should fit snugly in the box so that it will not roll or slide and suffer additional breakage.

5. A wooden block marked with the appropriate depth and run number is placed between each separate core run. In addition, wherever core is lost due to the presence of a cavity or large discontinuity (open or filled), a spacer is placed in the proper position in the core box. The spacer should be marked with the missing length and depth range along with the reason for the missing core; e.g., cavity, large joint, etc.
6. The core box is marked on the top and two ends with the client's name, site identification, boring number, depth range, and box number.

## **RECORDS AND DOCUMENTATION**

1. All data, sampling information, and rock classification will be recorded on rock coring logs (attached). Specifically, the following items will be included:
  - a. Type of core drill, including size of core.
  - b. Length of core recovered for each length drilled.
  - c. Depth at which rock was encountered.
  - d. Depth of bottom of hole.
  - e. Description of rock.
  - f. Time required to drill each foot.
  - g. Date, job name and number, drilling inspector.
  - h. Fluid loss information (amount or rate and depth interval).

### **SPECIAL NOTES**

1. Double-tube, diamond-bit core barrel and drilling tools will be cleaned via a high pressure hot water or steam rinse upon completion of the borehole to preclude cross-contamination between successive boreholes.
2. Wash water used during the core drilling will not be recirculated to the borehole unless specifically authorized by the drilling inspector.

### **APPLICABLE REFERENCES AND STANDARDS**

Department of the Navy, NFEC, Soil Mechanics, Design Manual 7.1, NAVFAC DM-7.1, Alexandria, Virginia, MAY 1982.

## **ROTARY DRILLING**

### **PURPOSE**

To produce a large diameter (six-inch [6"]) borehole in the bedrock at specified locations and to predetermined depths for testing of the hydrogeologic characteristics of the rock (pressure permeability, geophysical logging) and installation of multilevel groundwater sampling devices.

### **EQUIPMENT AND MATERIALS**

- Hydraulic or air rotary drilling rig with appropriate drilling tools
- Rotary drilling bit (roller bit) or pneumatic "down-the-hole" hammer

### **PROCEDURES**

1. At specified borehole locations, drill through overburden to top of rock surface.
2. Install steel/PVC casing from just above ground surface to rock surface by hydraulic pressure, driving and/or spinning to prevent collapse of the borehole in the overburden.
3. Advance borehole into bedrock to the required depth by hydraulic or air rotary drilling. Hydraulic rotary drilling consists of cutting the borehole by means of a rotating bit and removing the cuttings by continuous circulation of a drilling fluid as the bit penetrates the formation materials. Air rotary drilling utilizes compressed air as the "drilling fluid," noted above.
4. Collect at ten-foot (10') intervals and save in clean glass jars drill cuttings from the top of the rock surface to the bottom of the borehole for comparison with rock core.
5. Drill cutting sample jars shall be labeled with the boring number and sample depth.

### **RECORDS AND DOCUMENTATION**

1. All data, sampling information, and rock classification will be recorded on test boring logs (sample attached). Specifically, the following items will be included:
  - a. Type of drilling bit, including size.
  - b. Elevation at which rock was encountered.

- c. Elevation at bottom of hole.
- d. Description of rock cuttings.
- e. Time required to drill each foot, including hydraulic feed pressure.

#### **SPECIAL NOTES**

- 1. Rotary drilling bit and drilling tools will be sprayed with high pressure water and allowed to air dry upon completion of the borehole to preclude cross-contamination between successive boreholes.
- 2. Drilling fluids will not be recirculated to the borehole.

#### **APPLICABLE REFERENCES AND STANDARDS**

Groundwater and Wells, published by Johnson Division UOP, Inc., Saint Paul, Minnesota, 1975.

## **WELL INSTALLATIONS - OVERBURDEN WELLS**

### **PURPOSE**

Overburden (unconsolidated formation) monitoring wells are installed to provide access to groundwater for sampling or in situ testing purposes and for measurement of groundwater elevations.

### **EQUIPMENT AND MATERIALS**

- PVC pipe, flush-joint threaded (The diameter and schedule of pipe to be used will vary depending upon the specific project and application. Schedule 80, 2-inch nominal diameter is the standard monitoring well size. Smaller diameter piping is frequently used for Geoprobe explorations.) Note that certain chemical conditions such as the presence of dense non-aqueous phase liquid (DNAPL) may require the use of materials other than PVC.
- PVC slotted wellscreen, flush-joint threaded, matching the schedule and diameter of the riser pipe (0.01-inch slots, also referred to as “10 slot” are the “standard” size, but slot size may vary based on subsurface conditions). Well screen should be supplied in plastic wrap to preclude inadvertent contamination prior to installation.

Sediment/silt or DNAPL trap matching the material, schedule and diameter of the well screen (optional).

- Bentonite clay (chips, pellets and/or powder)
- Clean silica sand (The filter sand should be sized based on the well screen slot size and the grain size distribution of the formation into which the well will be installed. For most sand deposits and 0.01-inch slot size, a 20-40 sand or #0 filter sand is appropriate.)
- Portland cement
- Concrete mix
- Steel protective casing, 4-inch- to 6-inch- diameter, five-foot (5') length, with cap and locking device
- Flush-mounted road box

## PROCEDURES

1. Drill to one foot below the designated well depth and prepare the drill casing for the well installation. For drive and wash or water rotary borings, thoroughly flush the casing with clean water to remove suspended drill cuttings. Record the volume of water lost to the formation in this process. For hollow stem auger installations, remove the center plug and measure the depth to the bottom of the hole to verify that no soil is inside the auger. For Geoprobe installations where a filter pack is desired, fill casing with water before driving out the expendable bottom plug or use a pre-packed well screen. (Note that the well casing outside diameter must be correlated with the drill casing inside diameter to allow for the installation of a filter pack and annular seal. In general, there should be at least 1-inch difference in the two diameters.) Install a sand filter below the proposed base of the wellscreen approximately one-foot (1') thick by pouring sand down the borehole while withdrawing the casing one-foot (1'). (Note: where a DNAPL collection trap is Measure and record the depth of the sand cushion.
2. Assemble and install the observation well pipe and screen. Well tip must be fitted with a threaded or slip-on plug. All pipe sections shall be connected by dry threading of the joints. No glue, solvents, or lubricating compounds shall be used to make up the connections. The well pipe assembly must be carefully lowered into the borehole to ensure centering of the well in the hole. After installation, the GZA field engineer and the drilling contractor will carefully measure the depth to the well tip and record the measurement on the well log.
3. Install a sand filter around the wellscreen to at least two feet (2') above the screen. Grain size of the sand shall be appropriate for the slot size of the screen (normally one hundredth of an [0.01"] inch). This should be accomplished by placing filter sand up into the casing and gradually withdrawing the casing while measuring the top of the sand and verifying that the well remains at the desired depth. If too much sand is placed in the annular space inside the casing, it may result in binding of the well and casing and extraction of the well with the casing. Conversely, allowing the sand level to drop below the bottom of the casing could impact the quality and continuity of the filter pack.
4. Withdraw the casing to the top of the sand pack while monitoring the filter sand level and install a bentonite clay seal approximately two feet (2') thick above the sand filter. Where the drill hole will remain open, install the bentonite below the bottom of the casing. If the bentonite must be installed within the drill casing, work in short (< 1 foot) increments to prevent binding of the well and drill casing.
5. Insert a tremie pipe to just above the top of the bentonite seal and backfill the remainder of the hole with grout until it flows at the surface. (Grout should consist of cement-bentonite, high solids bentonite, or expansive cement mixtures to be selected by the drilling inspector.



Where the formation materials are relatively pervious, conventional Portland cement grout or an alternative annular seal, as outlined below, may be used.)

6. Square cut the well pipe at the top, as appropriate for the well completion method. For a flush-mount installation, the well casing should be set at an appropriate distance below the top of the road box to accommodate the locking well cap (usually a “gripper” plug, which requires about 0.2 feet of space.) For above-grade protective casings, the well pipe should be cut just below (within 0.1 feet) the level of the protective casing.
7. Install the protective casing or flush-mount road box into the borehole over the well casing. Make certain the well casing is securely capped during the road box or protective casing installation to avoid entry of soil or debris into the well. Complete the installation by constructing a concrete surface pad around the steel guard pipe or road box. The concrete pad shall be a minimum of 6 inches thick and 1.5 feet in diameter, sloped away from the protective casing/road box. For flush-mount installations, make certain the road box does not extend above the surface of the concrete pad. Record the precise distances from ground surface to the top of the well casing and road box/protective casing on the well log.

## RECORDS AND DOCUMENTATION

Well installations will be recorded on the drilling log for the hole. Installation details to be recorded include total well depth, screen depth and length, filter and seal depths and thicknesses, well head completion type, distances from ground surface to the well casing and road box or protective casing lip, distances from the well to fixed site features or GPS coordinates and any other details or measurements deemed necessary by the field engineer. All measurements should be made from ground surface.

## SPECIAL NOTES

1. Steel protective pipe or road boxes shall be steam cleaned or hot water power rinsed prior to installation to remove cutting oil or other residue. To assist in removal for sampling when using protective casings with threaded caps, the cap threads may be lubricated with a small amount of non-petroleum based material (e.g., vegetable oil).
2. If well is to be installed above the base of the borehole, care must be taken to ensure any underlying strata are properly sealed to preclude the possibility of cross contamination. Seals can be constructed using either grout or bentonite chips/pellets.
3. Grout backfill above the bentonite seal may be omitted under certain stratigraphic conditions. In those cases, the borehole should be backfilled with native materials with

occasional thin bentonite seals. Completed installation must have a vertical permeability less than that of the natural strata encountered.

4. When installing wells in fine-grained formations or where evaluation of hydraulic conductivity is a key objective, conventional filter sand may not be adequate. In these situations, the filter pack should be specifically designed in accordance with standard water well design practices.

#### **APPLICABLE STANDARDS AND REFERENCES**

U.S. Environmental Protection Agency, "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities," SW-611, December 1980.

## **WELL INSTALLATIONS - BEDROCK WELLS**

### **PURPOSE**

Bedrock wells are installed to provide access to groundwater contained in bedrock for sampling and monitoring purposes.

### **EQUIPMENT AND MATERIALS**

- Schedule 40 PVC pipe, one and one-half-inch (1-1/2") or two-inch (2") diameter, flush-joint threaded
- Schedule 40 PVC slotted wellscreen, one and one-half-inch (1-1/2") diameter, zero one hundred of an inch (0.01") slots, flush-joint threaded
- Clean silica sand
- Three-inch (3") or four-inch (4") diameter steel pipe, five-foot (5') length, threaded one end with cap and locking device
- Valve gate box
- Bentonite clay (pellets and powder)
- Cement
- Concrete mix
- Tremie pipe

## PROCEDURES

1. Install a one-foot (1') sand filter below the proposed base of the well screen.
2. Assemble and install the observation well pipe and screen in the required configuration for the hole. Well tip must be fitted with a threaded or slip-on bottom plug. All pipe connections shall be connected by dry-threading of the joints. No glue, solvents, or lubricating compound shall be used to make up the connections. The well pipe assembly must be carefully lowered into the borehole to ensure centering of the well in the hole. After installation, the GZA field engineer and the drilling contractor will carefully measure the depth to the well tip.
3. Install a sand filter around the well screen to at least one foot (1') above the screen. Grain-size of the sand shall be appropriate for the slot size of the screen (normally one hundred of an inch [0.01"]).
4. Install a bentonite clay seal (minimum two feet [2'] thick) above the sand filter.
5. Insert a tremie pipe to just above the top of the bentonite seal and backfill the remainder of the hole with grout until it flows at the surface. (Grout should consist of cement-bentonite, high solids bentonite or expansive cement mixtures to be selected by the drilling inspector.)
6. Square cut the well pipe approximately two feet (2') above grade.
7. Install a five-foot (5') section of steel pipe (three-inch [3"] or four-inch [4"] diameter) equipped with a threaded, lockable cap three feet (3') into the borehole. Complete the installation by constructing a concrete surface pad around the steel guard pipe. The concrete pad shall be a minimum of one-half-foot (1/2') deep and one and one-half-foot (1-1/2') in diameter and sloped away from steel casing.
8. If well-head completions must be flush with the ground surface, a street box or lockable valve gate box may be installed in lieu of the metal pipe. Installation consists of square cutting the well riser pipe two inches (2") below grade, and cement grouting the box in place.

## **RECORDS AND DOCUMENTATION**

Well installations will be recorded on the drilling log for the hole. Installation details to be recorded include total well depth, screen depth and length, filter and seal depths and thicknesses, well head completion type, and any other details or measurements deemed necessary by the field engineer.

## **SPECIAL NOTES**

1. Steel protective pipe or valve gate boxes shall be steam cleaned or hot water power-rinsed prior to installation to remove cutting oil or other residue. To assist in removal for sampling, the cap threads may be lubricated with a small amount of non-petroleum based material (vegetable oil or Crisco).
2. If well is to be installed above the base of the borehole, care must be taken to ensure any underlying strata are properly sealed to preclude the possibility of cross contamination. Seals can be constructed using either cement bentonite grout or bentonite pellets.
3. Grout backfill above bentonite seal may be omitted under certain stratigraphic conditions. In those cases, hole should be backfilled with native materials with occasional thin bentonite seals. Completed installation must have a vertical permeability less than that of the natural strata encountered.

## **APPLICABLE STANDARDS AND REFERENCES**

U. S. Environmental Protection Agency, "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities," SW-611, December 1980.

## WELL DEVELOPMENT

### PURPOSE

To enhance the hydraulic connection between the wellscreen and the overburden soil by removing fine soil materials from and rearranging the sand filter pack around the wellscreen.

### EQUIPMENT AND MATERIALS

- Centrifugal pump and tubing
- Container (e.g., 5-gallon bucket)
- Surge block and stem

### PROCEDURES

1. For well locations where static water levels are shallow (less than 25 feet [25'] deep), recharge is sufficiently rapid to prevent drawdown below 25 feet (25'), and groundwater is not significantly contaminated, "overpumping" is an advantageous well development method. For most well locations, centrifugal pumping is the best method; where centrifugal pumping is not feasible (e.g., low recharge), alternate purging methods may be used, such as inertial, gas-driven, or bladder pumps. The following procedure is followed:
  - a. Prior to well evacuation, lower a weighted tape to the bottom of the well. Compare the measured well depth to the depth of the well upon installation to assess if any fine material has accumulated in the bottom of the well.
  - b. Lower the pump tubing to the bottom of the well. Evacuate the well by pumping at a rate faster than the well would normally be pumped or purged for sample collection. Pump until discharge water, collected in a container, is visually clear of significant suspended sediment, or until the content of suspended sediment appears to stabilize after significant pumping. Tubing should be periodically raised and lowered within the well during pumping.
2. If overpumping alone does not appear to evacuate accumulated fine sediment, then this method may be combined intermittently with mechanical surging, which may free collected sediment from the filter pack and wellscreen. The following procedure is followed:
  - a. After attempts to overpump, slowly lower the surge block to the bottom of the well, increasing the speed of lowering and retracting near the bottom of the wellscreen. Care should be taken not to compact accumulated sediment or force it out through the wellscreen.

- b. Continue to intermittently surge and overpump the well; discontinue as in 2.A.

#### **APPLICABLE REFERENCES AND STANDARDS**

Massachusetts Department of Environmental Protection, Standard References, "Well Development" (Section 4.5), March 1989.



## **SAMPLE COLLECTION - SURFACE WATERS**

### **PURPOSE**

To obtain liquid samples for analyses that are representative of environmental conditions at the location sampled.

### **EQUIPMENT AND MATERIALS**

- Appropriate sample containers
- Pond sampler with disposable sampling containers
- Peristaltic pump with associated tubing
- Rags or terry cloths

### **PROCEDURES**

#### **1. Method A - Container Sampling**

This methodology will be utilized in flowing waters where minimum sample agitation is required, the sampling depth is less than one-foot (1'), and direct contact with the liquid being sampled will not endanger the sampling team.

- a. Approach the sampling point from downstream, submerge the sample container to the required depth upstream of the sampling platform (boat, etc.) and remove it after complete filling.
- b. Cap and seal the container
- c. Rinse and wipe off the exterior of the container with clean paper towels.
- d. Label, preserve, and store the sample in accordance with appropriate protocols.

Note: This method cannot be employed with prepreserved containers. Appropriately cleaned containers without preservative can be used to collect the sample for transfer to the preserved containers in this case.

## 2. Method B - Pond Sampler

This methodology will be utilized when contamination of the outside of the sample container must be avoided, the sampling depth is less than one-foot (1'), and direct contact with the liquid being sampled could endanger the sampling team.

- a. After arrival at the sampling point, assemble the pond sampler or similar sampling device and fit it with a disposable plastic beaker.
- b. Extend and submerge the sampler to the required depth (upstream of the sampling platform, if applicable), and remove it after complete filling.
- c. Carefully transfer the liquid from the sampler to the appropriate sample container by pouring the liquid down the side of the container with minimal turbulence. This procedure is critical to minimize loss of volatile materials from the sample through aeration.
- d. Cap and seal the sample container.
- e. Remove the disposable beaker from the sampling rod and dispose of it in a trash bag.
- f. Wipe down the sampling rod with clean rags. Store the sampling rod in plastic bags for subsequent cleaning. Dispose of the rags in a trash bag.
- g. Label, preserve, and store the sample in accordance with appropriate protocols.

## 3. Method C - Peristaltic Pump

This methodology will be utilized when the sampling depth is greater than one-foot (1'), but less than twenty-five feet (25'), and/or direct contact with the liquid being sampled would endanger the sampling team.

- a. Install clean, medical-grade silicon tubing on the pump head, allowing sufficient length on the discharge side for filling of sample containers.
- b. Select and install the necessary length of clean intake tubing to provide the required sampling depth. Intake tubing shall be heavy-wall Teflon of a diameter equal to that of the pump tubing.
- c. Submerge the intake tubing to the required depth by tying it off to a graduated, weighted cable or rope.

- d. Actuate the sample pump and allow two to three liters (2-3 l) of sample to purge the system before collection.
- e. Obtain sample by allowing pump discharge to gently flow down the side of the container with minimum turbulence. This procedure is critical to minimize loss of volatile materials from the sample through aeration.
- f. Cap and seal the sample container.
- g. Stop pump, allow system to drain, then disassemble.
- h. Place tubing in plastic bag and return to Newton laboratory for decontamination.
- i. Label preserve, and store the sample in accordance with appropriate protocols.

## **RECORDS AND DOCUMENTATION**

All data and sampling information will be recorded as specified in the Quality Assurance Project Plan.

## **SPECIAL NOTES**

- 1. Samples for oil and grease analysis should be collected using Method A, as significant amounts of material could adhere to the sample transfer container used in Method B.
- 2. Method A has significant shortcomings in sampling concentrated or high hazard waste streams, due to physical contact with the waste stream and contamination of the outside of the sample container.
- 3. Required depth for surface water sampling will be one-half the total depth of the water body (mid-depth) unless otherwise specified.

## **APPLICABLE REFERENCES AND STANDARDS**

deVera, E.R.; Simmons, B.P.; Stephens, R.D.; and Storm, D.L., "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA-600/2-80-018, January 1980.

U.S. Environmental Protection Agency, "Procedures for Groundwater Monitoring at Solid Waste Disposal Facilities. EPA-530/SW-611, August 1977.

Ford, J.J.; Turina, P.J.; and Seely, D.E., "Characterization of Hazardous Waste Sites - A Methods Manual, Volume II, Available Sampling Methods, EPA 600/X-83-018, March 1983.

## **SAMPLE COLLECTION - MONITORING WELLS**

### **PURPOSE**

To obtain representative samples of groundwater from designated monitoring well locations for chemical analysis.

### **EQUIPMENT AND MATERIALS**

- Well keys and/or road box wrench
- Appropriate sample containers
- Peristaltic pump with ¼" polyethylene tubing and tygon tubing for pump head, bladder pump with associated controller and tubing or disposable bailers with polypropylene cord
- One hundred-foot (100') fiberglass tape with weighted end
- Field parameter meter
- Cooler and ice
- Electric water level reader or interface probe
- Flow measurement device
- Power source
- Decontamination supplies (clean water, paper towels, etc)
- Health and safety equipment (gloves and other relevant gear)

Additional equipment and materials such as field screening equipment, filtering equipment, etc., may be needed for particular sampling rounds.

### **PROCEDURES**

1. Confirm details of sampling program (locations, parameters for analysis, etc.) and laboratory coordination (receipt of bottle order). Review well installation logs for information on well construction including well depth, diameter, and screened interval. Plan sampling sequenced based on previous analytical data, if available. Sampling should proceed from cleaner to more contaminated locations, as appropriate.
2. Check well conditions (protective casing/road box integrity, presence of locking cap) and record significant observations. Confirm well identifications prior to initiating sample collection.
3. Open well casing and measure depth to groundwater to the nearest one-hundredth of a foot (0.01') using an electrical water level indicator. If a light non-aqueous phase liquid (LNAPL) is anticipated, use an interface probe in place of the water level reader to measure the water level and LNAPL thickness. If reliable information on well depth is not available,

measure total well depth using a weighted tape (see note below).

4. Select appropriate well purging and sampling method (from items A through C below) based on project objectives, well construction details and site background. In general the modified low-flow sampling protocol will be the appropriate sample collection approach, although project-specific considerations may dictate the selection of an alternative method.

**A. MODIFIED LOW-FLOW SAMPLING PROCEDURE (PERISTALTIC PUMP)**

1. Insert 1/4"-polyethylene tubing to the midpoint of the screened interval of the well. If dedicated tubing is present in the well, adjust tubing depth to place intake at midpoint of screened section of well.
2. Connect 1/4" polyethylene tubing to the peristaltic pump intake line and route discharge to the flow-through cell of the field meter, or to a small container. Select ultimate discharge location for purge water (see notes below) and route discharge line from flow-through cell or container to the selected location.
3. Turn on peristaltic pump and adjust flow to approximately 0.1 to 0.5 liters per minute (lpm). Check flow rate with graduated container.
4. Monitor and record the following field parameters at regular intervals (3 to 15 minutes):
  - Dissolved Oxygen (DO)
  - Specific Conductance (SC)
  - Temperature (T)
  - pH
  - Oxidation/Reduction Potential (ORP-Eh)

For many situations, turbidity should also be measured at these same intervals.

5. Check water level in well and adjust pumping rate to limit drawdown.
6. Collect groundwater samples for chemical analyses upon stabilization of the field parameters. (Note that a minimum volume equal to the standing volume in the extraction tubing plus the stabilized drawdown volume within the well casing must be removed before sample collection can begin, even if field parameters stabilize. For 2-inch diameter wells less than 100 feet deep, stabilized drawdowns less than 5 feet and 1/4-inch extraction tubing, this minimum volume will be less than 1 gallon.). Disconnect pump discharge from flow-through cell and route directly to the sample container. Stabilization is defined as consecutive readings, at least three minutes apart, which vary within the following limits.

DO	-	10%
SC	-	10%
Temperature	-	5%
pH	-	0.2 units
ORP	-	20 millivolts

(Note that these stabilization parameters were developed from the United States

Environmental Protection Agency Region 1 guidance, modified by GZA's field experience. These limits should be considered as approximate guidelines only.)

7. If metals analyses are planned, measure and record turbidity upon stabilization of the other parameters. Turbidity readings of less than 5 NTU are desirable for low-flow samples. If turbidity exceeds this value consider field filtration of samples. Collect filtered samples by connecting peristaltic pump discharge directly to the upstream end of a 0.45 micron cartridge filter. Purge approximately one volume of the cartridge filter before collecting sample.

#### **B. MODIFIED LOW-FLOW SAMPLING – SUBMERSIBLE PUMP.**

1. Connect submersible pump (generally a bladder pump or electrical pump) to discharge line and controller and lower intake to midpoint of well screen.
2. Connect pump discharge to the flow-through cell of the field meter or route to a small container, suitable for manual measurements. Select ultimate discharge for purge water (see notes below) and route discharge line appropriately.
3. Turn on the pump and adjust flow to approximately 0.1 to 0.5 lpm. Check flow rate with a graduated cylinder.
4. Follow steps 4 through 7 under Section A above.

#### **C. DISPOSABLE BAILER/PERISTALTIC PUMP SAMPLING**

1. Determine standing well volume based on well depth and measured water level.
2. Purge 3 to 5 standing well volumes using either a new disposable bailer, a bailer dedicated to the well, a pump with dedicated or disposable intake components (e.g. a peristaltic pump with intake tubing dedicated to the well or an inertial pump with a dedicated or disposable foot valve and tubing). If re-usable purging equipment is employed (e.g. an electrical submersible pump), the pump must be thoroughly decontaminated between monitoring wells. See notes below.
3. After the calculated purge volume has been extracted, remove pumping equipment from the well (as appropriate). For bailer sampling, lower the bailer slowly to the midpoint of the well screen interval, then retrieve it and transfer the groundwater to the appropriate sample containers. Pour liquid from the bailer down the side of the container to minimize the turbulence. For peristaltic pump sampling, adjust the intake tubing depth to the midpoint of the well screen, then turn on pump and adjust flow to 0.1-0.5 lpm. Route pump discharge line to appropriate sample containers in a manner that minimizes turbulence.
5. Collect samples destined for volatile organic compounds (VOC) analysis first. Make certain that the glass vials are completely filled, but do not overfill (vials are generally preserved in advance with HCl). Check for air bubbles after filling vial. If air bubbles are noted, discard the vial and fill another empty vial. Label vials, or confirm label information, and place vials into an ice-packed cooler for transport to the laboratory.



6. Collect additional samples, as required, in accordance with the container and preservation requirements.
7. Collect duplicate samples, matrix spikes or equipment blank samples as required by the project-specific sampling plan.
8. Clean exterior of the sample containers with clean water and paper towels prior to placing them into the cooler for transport to the laboratory.
9. Secure the well cap and road box or protective casing upon completion of sampling at each location.
10. Complete a chain-of-custody sheet daily for each sampling round.

## **RECORDS AND DOCUMENTATION**

Record water levels and well condition information in a field book or data form. Record any relevant observations such as the presence of odors or sheens. If the low-flow sampling technique is employed, record water quality data on the sampling form. Complete a chain of custody form including relevant sample collection information.

## **SPECIAL NOTES**

1. If LNAPL is present within a well, collection of groundwater samples for analysis of dissolved parameters is generally not appropriate. Specialized sampling techniques should be employed where analysis of the groundwater below separate phase layer is required.
2. Most electrical water level readers are not designed for prolonged submersion below the water table and should generally not be used for measuring well depth. Additionally, any equipment used to measure well depth should be thoroughly decontaminated between wells.
3. Disposition of purge water associated with groundwater sampling rounds must be evaluated prior to initiation of sampling. In Massachusetts, the Massachusetts Contingency Plan (310 CMR 40.0045(7)) allows for discharge of purge water to the surface in the immediate area of the well. This can be accomplished by discharge to the ground adjacent to the well, or store the purge water in a container temporarily and then pouring the water back into the well. Care must be taken to avoid discharging sediments to the well. In some situations, purge water must be collected for appropriate treatment/disposal.

## **APPLICABLE REFERENCES AND STANDARDS**

U.S. EPA, Region 1, "Low Stress (low flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells", SOP# GW0001, July 30, 1996

U.S. EPA, "Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater", September 1988

Massachusetts Department of Environmental Protection, “Standard References for Monitoring Well Installations”, Section 6.2 – “Sampling Techniques”, January 1991

## **SAMPLE COLLECTION - SURFACE SOIL**

### **PURPOSE**

Surface soil samples are collected to determine the physical characteristics of the material or levels of contamination in the unsaturated zone.

### **EQUIPMENT AND MATERIALS**

- Appropriate sample containers
- D-handle shovel
- Disposable polypropylene scoops
- T-handle auger
- Split-spoon sampler with extension rods and T-handle
- Stainless steel laboratory spoons
- Clean rags or terry cloths

### **PROCEDURES**

#### **Method A - Scoop Sampling**

This methodology will be utilized to collect soil samples from depths of less than one-foot (1') when exact sampling depth within the one-foot (1') interval is not critical.

1. Clear away all surface debris (leaves, twigs, etc.) for a one-foot (1') radius around the sampling location.
2. Using a precleaned D-handle shovel or soil trowel, excavate the soil to the desired sampling depth and stockpile the material on a portion of the cleared area.
3. Transfer material from the stockpile to a suitable sample container with a polypropylene scoop or steel soil trowel.
4. Cap and seal the container.
5. Label, preserve, and store the container in accordance with the Quality Assurance Project Plan.
6. Place the polypropylene scoop, if used, in a trash bag for subsequent disposal.
7. Wash the shovel or trowel with methanol, followed by distilled water, and dry with clean

paper towels prior to subsequent sampling.

#### Method B - Spoon Sampling

This methodology will be utilized to collect soil samples from depths of less than one-foot (1') when exact sampling depth within the one-foot (1') interval is critical.

1. Clear away all surface debris (leaves, twigs, etc.) for a one-foot (1') radius around the sampling location.
2. Using a precleaned D-handle shovel, excavate the soil to the desired sampling depth.
3. Obtain a sample from the excavation hole sidewall at the desired sampling depth using a stainless steel laboratory spoon and transfer it directly to a suitable sample container.
4. Cap and seal the container.
5. Label, preserve, and store the sample in accordance with the Quality Assurance Project Plan.
6. Wash the shovel and spoon with methanol, followed by distilled water, and wipe it down with clean paper towels prior to subsequent sampling.

#### Method C - Split-Spoon Sampling

This methodology will be utilized to collect soil samples down to a depth of approximately six feet (6') when soil conditions allow.

1. Clear away all surface debris (leaves, twigs, etc.) for a one-foot (1') radius around the sampling location.
2. Assemble a precleaned auger and auger a hole to the desired sampling depth. Carefully withdraw the auger to prevent cave-in of the borehole sidewalls.
3. Assemble a precleaned split-spoon sampler with appropriate extension rods and T-handle. Insert the sampler into the hole and force it into the soil with a twisting motion. Carefully withdraw the sampler and disassemble it.
4. Discard the upper one-inch (1") of the sample. Remove the remainder with a clean stainless steel laboratory spoon and transfer it directly to a suitable sample container.
5. Label, preserve, and store the sample in accordance with the Quality Assurance Project

Plan.

6. Place the sampling equipment in a plastic trash bag for subsequent cleaning.

## **RECORDS AND DOCUMENTATION**

All data and sampling information will be recorded as specified in the Quality Assurance Project Plan.

## **SPECIAL NOTES**

None

## **APPLICABLE STANDARDS AND REFERENCES**

Dunlap, W.J.; McNabb, J.F.; Scaf, M.R.; and Crosby, R.L., "Sampling for Organic Chemicals and Microorganisms in the Subsurface," EPA-600/2-77-16, August 1977.

U.S. Environmental Protection Agency, "Characterization of Hazardous Waste Sites - A Methods Manual, Volume II - Available Sampling Methods, EPA 600/X-83-018, March 1983.

## **SAMPLE COLLECTION - SUBSURFACE SOIL**

### **PURPOSE**

Subsurface soil samples are collected to determine the physical characteristics or levels of contamination of overburden material at any desired depth.

### **EQUIPMENT AND MATERIALS**

- Appropriate sample containers
- Split-spoon sampler with appropriate drill rods
- Drill rig with one hundred-forty-pound (140-lb) drive weight
- Stainless steel laboratory spoons
- Clean rags or terry cloths

### **PROCEDURES**

1. After boring and clean-out of the hole to the desired sampling depth, assemble the sampler and lower it carefully to the bottom of the hole.
2. With the split spoon sampler set at the bottom of the hole (be sure sampler is at the bottom of the casing), the drill rod should be marked at three consecutive six-inch (6") intervals for measuring the blows per six inches (6") of driving. If the sampler is above the bottom of the casing, this indicates that the bottom has been disturbed and soil has risen up the casing. Do not attempt to sample. Withdraw the sampler and clean the hole. If soil has blown up the casing, the casing should be advanced to below the disturbed soil. Clean-out alone is not sufficient. Follow the instructions in Step 6 of this section.
3. The sampler should be driven by a one hundred-forty-pound (140-lb) weight falling freely thirty inches (30"). Check to make sure that the fall is thirty inches (30") by marking the drive head and that the hammer is falling freely. The driller should use no more than two wraps of the rope around the cathead. Be certain that the rope is fully released to permit complete free fall of the hammer. The number of blows to drive each six-inch (6") interval should be recorded. The sampler should be driven at least eighteen inches (18"), unless the blow count exceeds one hundred (100) blows per six inches (6") or unless refusal, as defined in the specification, is met. When the blow count exceeds one hundred (100) blows per six inches (6"), the driving may be stopped and the sampler removed. The number of blows and the inches penetrated should be recorded. After driving in dense soils, the drill rods may have to be turned clockwise to free the sampler for removal. Turning counterclockwise will only loosen the joints, and you may lose the sampler in the hole. Bumping up the rods should be avoided, if possible, because it tends to reduce the amount of soil recovery.

However, it is sometimes necessary in very dense soils in order to free the sampler.

4. In most cases, but especially when sampling in sands and loose silts, the driller should keep the casing full of water at all times. This will require adding water while the rods are being withdrawn prior to sampling and when sampler is withdrawn. If this measure is not sufficient to prevent soil from running up into the casing, then commercial drilling mud should be used. Use a perforated section of drill rod above the sampler to facilitate drainage of drill fluid after sampling. When sampling soils for chemical testing, however, no water should be used so that the geochemical integrity of the sample is preserved.
5. When the sampler is brought to the ground surface, it should be opened immediately, and the length of recovery should be measured and recorded. Any loose wash at the top of the sample should not be counted as part of the recovery. If recovery is less than six inches (6"), another sample should be taken immediately below this sample, except in certain instances, such as where rock is encountered and coring is necessary.
6. If recovery is insufficient, put sampler back down hole and proceed as follows: if original depth is reached, drive sampler eighteen inches (18") and record blows as new sample; if original depth is not reached, redrive sampler to recover disturbed material record only original blow count and that sample recorded or redrive.
7. Remove sample with a clean laboratory spoon and transfer it directly to a suitable sample container.
8. Label, preserve, and store the sample in accordance with the Quality Assurance Project Plan.
9. Wash the split spoon with water and rinse with reagent grade methanol followed by distilled water.

## **RECORDS AND DOCUMENTATION**

1. All sampling information will be recorded as specified in the Quality Assurance Project Plan.
2. Penetration resistance will be recorded on standard boring log forms, pages 1 and 2.

## **SPECIAL NOTES**

None

## **APPLICABLE STANDARDS AND REFERENCES**



ASTM D1586-67

## **SAMPLE COLLECTION - LAKE, POND, AND STREAM SEDIMENTS**

### **PURPOSE**

Sediment samples underlying water bodies are collected to describe the physical characteristics of the sediment or to investigate contamination in the sediments.

### **MATERIALS AND EQUIPMENT**

- Appropriate sample containers
- Pond sampler with disposable collection jars
- Hand corer
- Eckman or Ponar grab
- Stainless steel laboratory spoon
- Stainless steel or polypropylene tray
- Clean rags or terry cloths
- Three-quarter-inch (3/4") nylon line

### **PROCEDURES**

#### **Method A - Hand Corer**

This methodology will be utilized in shallow (less than three feet [3']) water depths when a relatively undisturbed sample is required or when samples of up to eighteen inches (18") depth are required. Teflon, brass, or stainless steel core liners may be used with this technique to prevent cross-contamination.

1. Position sampling platform downstream of sample collection point.
2. Assemble a precleaned hand corer by attaching core tube to drive head or inserting core liner into assembly.
3. Insert the corer through the water column into the sediment and force it in with a smooth pressure.
4. Twist the corer approximately ninety degrees (90°) and slowly withdraw it in a single motion.
5. Either extrude the tube or remove the nosepiece and withdraw the sample into a stainless steel or aluminum tray.

6. Transfer the sample to a suitable sample container using a stainless steel laboratory spoon.
7. Cap and seal the sample container.
8. Flush the corer with clean water to remove any residual sediment.
9. Wash the sample tray with methanol and distilled water and wipe it down with clean paper towels.

#### Method B - Pond Sampler

This methodology will be utilized in shallow (less than three feet [3']) water depths when disturbance of the sediment by the sampling technique will not affect sample integrity.

1. Position sampling platform (boat, etc.) downstream of sample collection point.
2. Assemble pond sampler by attaching a polypropylene collection jar to the end and extending the handle to the required length.
3. Insert the sampler through the overlying water with the collection jar facing downward.
4. When contact with the sediment is felt, rotate the sampler one hundred and eighty degrees (180°) while exerting downward pressure to obtain a sample.
5. Slowly withdraw the sampler and place the sample in a stainless steel or aluminum tray.
6. Transfer the sample to an appropriate sample container using a stainless steel laboratory spoon.
7. Cap and seal the sample container.
8. Wash the sample tray with methanol and distilled water and wipe it down with clean paper towels.
9. Remove the collection jar from the sampler and place it in a trash bag for subsequent disposal.

### Method C - Grab Sampler

This methodology will be utilized in water depths exceeding three feet (3').

1. Attach a pre-cleaned Ponar or Eckman grab to an appropriate length of three-quarter-inch (3/4") nylon line.
2. Open grab jaws and set the release mechanism. Keep tension on nylon line to prevent premature tripping of the jaws.
3. Slowly lower the grab through the water column until contact with the sediment is felt.
4. Allow nylon line to slack approximately one-foot (1'), then slowly remove the slack and raise the grab to the surface.
5. Place the grab in a stainless steel or polypropylene tray, open the jaws to release the sample, and remove the grab from the tray.
6. Transfer the sample from the tray to a suitable sample container using a stainless steel laboratory spoon.
7. Cap and seal the sample.
8. Flush the grab with clean water to remove any residual sediment.
9. Wash the sample tray with methanol and distilled water and wipe it down with clean paper towels.

### RECORDS AND DOCUMENTATION

Samples will be labeled, preserved, and stored in accordance with the Quality Assurance Project Plan.

### SPECIAL NOTES

None

## **APPLICABLE STANDARDS AND REFERENCES**

Ford, P.J.; Turina, P.J.; and Seely, D.E., "Characterization of Hazardous Waste Sites - A Methods Manual," Volume II - Available Sampling Methods, EPA 600/S-83-018, 1983.

## **CHAIN-OF-CUSTODY RECORD KEEPING**

### **PURPOSE**

A chain-of-custody record is kept to provide documentation linking the collection of a sample to the performance of a laboratory test on that sample. That documentation includes: the name of the sample collector, the type of sample, the location of the sampling, the date and time of the sampling, and the names of the individuals who transported the sample from the field to the laboratory.

### **EQUIPMENT**

GZA or other project approved chain-of-custody record form.

### **PERFORMANCE**

1. Review the project work plan and the project contract for specific chain-of-custody record keeping requirements. Requirements detailed in either the project work plan or the project contract take priority over the instructions detailed below.
2. The chain-of-custody form is shipped inside the transporting container. If the samples are shipped by public courier (if Federal Express, UPS, etc.) the airbill generally serves as the chain-of-custody record for that portion of the trip.
3. The field drilling log generally serves as the chain-of-custody record for transportation of split spoon samples from the field to GZA's laboratory. A separate chain-of-custody record form must be used to transport soil samples to outside laboratories.
4. The intent is to use one (more as required) GZA chain-of-custody record form per day.
5. In general, one line on GZA's chain-of-custody form can suffice for each station number. When more than one sample is collected at a station, use the following:

Time - Provide the time the sampling was completed at that station.

Container ID - For this situation this column is non-applicable and should be so indicated (i.e., NA).

Sampler ID - If more than one sampling tool is used, indicate the number used and describe each on the record notes.

Note that project QA/QC procedure may require that each sample container be tracked.

This will necessitate the use of a separate line for each container.

6. Prior to leaving the site, verify that all pertinent data is on the chain-of-custody form.

#### **RECORDS AND DOCUMENTATION**

1. The white original is forwarded to the laboratory. That copy should be returned to GZA with the laboratory results.
2. The yellow copy is retained by the collector. That copy is to become part of that day's field records.
3. The pink copy is returned to the Project Manager for inclusion in project files.

#### **SPECIAL NOTES**

None

#### **APPLICABLE REFERENCES AND STANDARDS**

GZA chain-of-custody record form.

(Chain-of-custody form)



**APPENDIX E**  
**EQUIPMENT USER MANUALS**

# MiniRAE 2000

## Portable VOC Monitor PGM-7600




## OPERATION AND MAINTENANCE MANUAL

(Document No.: 011-4001-000)  
Revision E, May 2005



## **ATTENTION!**

### **For European Applications**

- A. CE 0575  II 1G/2G  
DEMKO 03 ATEX 0204759X  
Eex ia IIC T4**
- B. Recharge batteries only in non-hazardous locations.**
- C. Do not connect external cable to serial interface jack in hazardous locations.**
- D. Use RAE Systems Adapter P/N 500-0072 for connection to communication port and charging jack only in a non-hazardous area.**

# Table of Contents

<b>1. GENERAL INFORMATION .....</b>	<b>1-1</b>
1.1 General Specifications .....	1-2
<b>2. OPERATION OF MINIRAE 2000 .....</b>	<b>2-1</b>
2.1 Physical Description .....	2-2
2.2 Keys and Display .....	2-3
2.3 Power On/Off .....	2-4
2.4 Operation.....	2-5
2.4.1 Survey Mode .....	2-6
2.4.2 Hygiene Mode .....	2-14
2.5 Alarm Signals.....	2-16
2.6 Preset Alarm Limits and Calibration .....	2-18
2.7 Integrated Sampling Pump.....	2-19
2.8 Back Light.....	2-20
2.9 Datalogging.....	2-21
<b>3. OPERATION OF ACCESSORIES .....</b>	<b>3-1</b>
3.1 Standard Kit and Accessories.....	3-2
3.2 Optional Accessories.....	3-5
<b>4. PROGRAMMING OF MINIRAE 2000 .....</b>	<b>4-1</b>
4.1 Programming Mode .....	4-2
4.2 Keys for Programming Mode .....	4-3
4.3 Entering into Programming Mode .....	4-4
4.4 Calibrate and Select Gas .....	4-5
4.4.1 Fresh Air Calibration.....	4-8
4.4.2 Span Calibration .....	4-9
4.4.3 Select Cal Memory .....	4-11
4.4.4 Change Span Value .....	4-12
4.4.5 Modify Cal Memory .....	4-13
4.4.6 Change Correction Factor.....	4-14

4.5	Change Alarm Limits .....	4-15
4.5.1	Change Low Alarm Limit .....	4-17
4.5.2	Change STEL Limit .....	4-18
4.5.3	Change TWA Limit .....	4-19
4.6	View or Change Datalog .....	4-20
4.6.1	Reset Peak/Minimum .....	4-21
4.6.2	Clear Data .....	4-223
4.6.3	Change Data Period .....	4-2324
4.6.4	Change Average Type .....	4-245
4.7	Change Monitor Setup .....	4-256
4.7.1	Change Operation Mode .....	4-267
4.7.2	Change Site ID .....	4-278
4.7.3	Change User ID .....	4-2829
4.7.4	Change Alarm Mode? .....	4-290
4.7.5	Change User Mode .....	4-301
4.7.6	Change Date .....	4-312
4.7.7	Change Time .....	4-323
4.7.8	Change Lamp .....	4-334
4.7.9	Change Unit .....	4-345
4.7.10	Change Dilution Ratio .....	4-356
4.7.11	Change Output? .....	4-367
4.7.12	Change DAC Range? .....	4-378
4.7.13	Set Temperature Unit? .....	4-389
4.8	Exit Programming Mode .....	4-390
<b>5.</b>	<b>THEORY OF OPERATION .....</b>	<b>5-1</b>
<b>6.</b>	<b>MAINTENANCE .....</b>	<b>6-1</b>
6.1	Battery Charging and Replacement .....	6-2
6.2	PID Sensor & Lamp Cleaning /Replacement .....	6-4
6.3	Sampling Pump .....	6-7
6.4	Turning on the UV Lamp .....	6-9
<b>7.</b>	<b>TROUBLESHOOTING .....</b>	<b>7-1</b>
7.1	Troubleshooting Table .....	7-2
<b>APPENDIX A. QUICK REFERENCE GUIDE .....</b>		<b>A-2</b>



## **WARNING**



**- Do NOT proceed before reading -**

**This manual must be carefully read by all individuals who have or will have the responsibility for using, maintaining, or servicing this product.**

The product will perform as designed only if it is used, maintained, and serviced in accordance with the manufacturer's instructions.

### **CAUTION!!**

**To reduce the risk of electric shock, turn off power before removing the monitor cover. Disconnect the battery before removing sensor module for service. Never operate the monitor while the cover is removed. Remove monitor cover and sensor module only in an area known to be non-hazardous.**

**The model PGM-7600 equipment is classified as to intrinsic safety for use in class I, division 1, groups A, B, C, D, or non-hazardous locations only.**

# Special Notes

-1-

**When the MiniRAE 2000 Monitor is taken out from the transport case and turned on for the first time, there may be some residual organic or inorganic vapor trapped inside the detector chamber. The initial PID sensor reading may indicate a few ppm. Enter an area known to be free of any organic vapor and turn on the monitor. After running for several minutes, the residual vapor in the detector chamber will be cleared and the reading should return to zero.**

-2-

**The battery of the MiniRAE 2000 monitor will discharge slowly even if it is turned off. If the monitor has not been charged for 5-7 days, the battery voltage will be low. Therefore, it is a good practice to always charge the monitor before using it. It is also recommended to fully charge the monitor FOR AT LEAST 10 HOURS before first use. See Section 7 for more information on battery charging and replacement.**

## **WARNINGS**

**STATIC HAZARD:** Clean only with damp cloth.

**For safety reasons this equipment must be operated and serviced by qualified personnel only. Read and understand instruction manual completely before operating or servicing.**

**Use only RAE Systems battery packs, part nos. 012-3050, 012-3051 or 012-3052. This instrument has not been tested in an explosive gas/air atmosphere having an oxygen concentration greater than 21%. Substitution of components may impair intrinsic safety. Recharge batteries only in non-hazardous locations.**

**The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service.**

**For maximum safety, the accuracy of the MiniRAE 2000 should be checked by exposing it to a known concentration calibration gas before each day's use.**



## **AVERTISSEMENTS**

**DANGER RISQUE D'ORIGINE ELECTROSTATIQUE:**  
**Nettoyer uniquement avec un chiffon humide.**

**Pour des raisons de sécurité, cet équipement doit être utilisé, entretenu et réparé uniquement par un personnel qualifié. Étudier le manuel d'instructions en entier avant d'utiliser, d'entretenir ou de réparer l'équipement.**

**Utiliser seulement l'ensemble de batterie RAE Systems, la référence 012-3050, 012-3051 au 012-3052. Cet instrument n'a pas été essayé dans une atmosphère de gaz/air explosive ayant une concentration d'oxygène plus élevée que 21%. La substitution de composants peut compromettre la sécurité intrinsèque. Ne charger les batteries que dans emplacements désignés non-dangereuse.**

**La calibration de tous les instruments de RAE Systems doit être testée en exposant l'instrument à une concentration de gaz connue par une procédure d'étalonnage avant de mettre en service l'instrument pour la première fois.**

**Pour une sécurité maximale, la sensibilité du MiniRAE 2000 doit être vérifiée en exposant l'instrument à une concentration de gaz connue par une procédure d'étalonnage avant chaque utilisation journalière.**

## 1. GENERAL INFORMATION

**MiniRAE 2000** Portable VOC Monitor (Model PGM 7600) is a compact monitor designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It monitors Volatile Organic Compounds (VOC) using a Photo-Ionization Detector (PID) with a 9.8 eV, 10.6 eV, or 11.7 eV gas discharge lamp. Features are:

- **Lightweight and Compact**
  - Compact, light weight (19 oz.) and rugged design
  - Built-in sample draw pump
- **Dependable and Accurate**
  - Up to 10 hours of continuous monitoring with rechargeable battery pack
  - Designed to continuously monitor VOC vapor at ppm levels
- **User Friendly**
  - Preset alarm thresholds for STEL, TWA, low and high level peak values. Audio buzzer and flashing LED display are activated when the limits are exceeded.
- **Datalogging Capabilities**
  - 15,000 point datalogging storage capacity for data download to PC


**MiniRAE 2000** consists of a PID with associated microcomputer and electronic circuit. The unit is housed in a rugged ABS + PC case with a backlit 1 line by 8 character dot matrix LCD and 3 keys to provide easy user interface.

## 1.1 General Specifications

Table 1.1

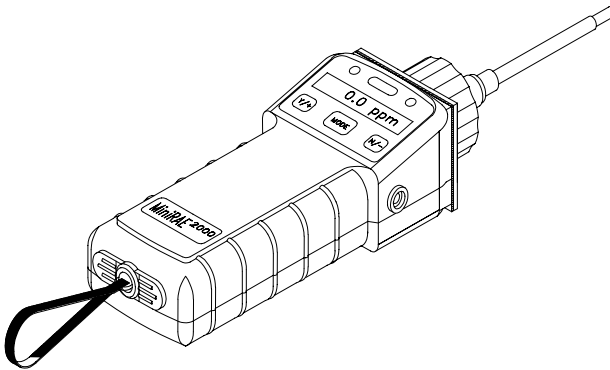
<b>Portable VOC Monitor Specification</b>	
Size:	8.2"L x 3.0"W x 2.0"H
Weight:	19.5 oz with battery pack
Detector:	Photo-ionization sensor with 9.8, 10.6, or 11.7 eV UV lamp
Battery:	A 4.8V /1250 mAH Rechargeable Nickel Metal Hydride battery pack (snap in, field replaceable)
Battery Charging:	10 hours charge through built-in charger
Operating Hours:	Up to 10 hours continuous operation
Display:	1 line by 8 characters 5x7 dot matrix LCD (0.4" character height) with LED back light automatically in dim light
Range, Resolution & Response time ( $t_{90}$ ):	
Isobutylene (calibration gas)	
	0-99 ppm                      0.1 ppm 2 sec
	100-1,999 ppm 1.0 ppm    2 sec
	2000-10,000 ppm 1.0 ppm 2 sec
Measurement Accuracy (Isobutylene):	
	0 – 2000 ppm: $\pm 2$ ppm or 10% of reading.
	> 2000 ppm: $\pm 20\%$ of reading
PID Detector:	Easy access to lamp and sensor for cleaning and replacement
Correction Factors:	Built-in 102 VOC gases
Calibration:	Two-point field calibration of zero and standard reference gas
Calibration Memory:	
	Store up to 8 separate calibration, alarm limits and span value
Inlet Probe:	Flexible 5" tubing
Keypad:	1 operation key and 2 programming keys

## GENERAL INFORMATION

Direct Readout:	Instantaneous, average, STEL and peak value, battery voltage and elapsed time
Intrinsic Safety:	UL & cUL Class 1, Division I, Group A,B,C,D, Temperature Code T3C (US & Canada); <b>CE</b> 0575  II 1G DEMKO 02 ATEX 0204759 Eex ia IIC T4 (Europe)
EM Interference:	No effect when exposed to 0.43 W/cm <sup>2</sup> RF interference (5 watt transmitter at 12 inches)
Alarm Setting:	Separate alarm limit settings for Low, High, STEL and TWA alarm
Operating Mode:	Survey or Hygiene mode
Alarm:	90 dB buzzer and flashing red LEDs to indicate exceeded preset limits, low battery voltage, or sensor failure.
External Alarm:	Optional plug-in pen-size vibration alarm or remote alarm
Alarm Mode:	Latching or automatic reset
Real-time Clock:	Automatic date and time stamps on data logged information
Datalogging:	15,000 points with time stamp, serial number, user ID, site ID, etc.
Communication:	Upload data to PC and download instrument setup from PC through RS-232 port
Sampling Pump:	Internally integrated. Flow rate: 450-550 cc/min.
Temperature:	0° to 45°C (32° to 113°F)
Humidity:	0 % to 95 % relative humidity (non-condensing)
Housing:	ABS + PC, conductive coating, splash and dust proof, will withstand 1 meter drop test with rubber boot
Attachment:	Wrist strap, rubber boot and belt clip

## 2. OPERATION OF MINIRAE 2000

The MiniRAE 2000 Portable VOC Monitor is a compact Monitor designed as a broadband VOC gas monitor and datalogger for work in hazardous environments. It gives real time measurements and activates alarm signals whenever the exposure exceeds preset limits. Prior to factory shipment the MiniRAE 2000 is preset with default alarm limits and the sensor is pre-calibrated with standard calibration gas. However, the user should test the instrument and verify the calibration before the first use. After the monitor is fully charged and calibrated, it is ready for immediate operation.



**Figure 2-1 MiniRAE 2000**

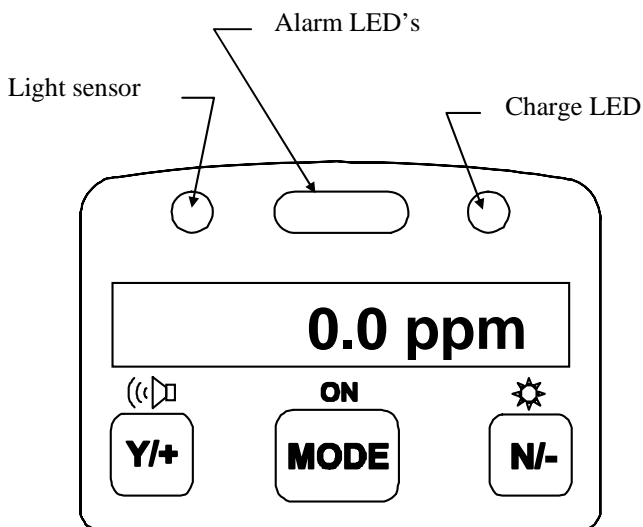
## **2.1 Physical Description**

The main components of the MiniRAE 2000 Portable VOC monitor include:

- Three keys for user to interact with the monitor: 1 operation key and 2 programming keys for normal operation or programming of the monitor
- LCD display with back light for direct readout and calculated measurements
- Buzzer and red LED's for alarm signaling whenever the exposures exceed preset limits
- Wrist strap
- Charge contact for plugging directly to the charging station
- Gas entry and exit ports
- Serial communication port for PC interface
- External alarm and analog output port
- Protective rubber cover

## 2.2 Keys and Display

Figure 2.2 shows the LCD display and the keypad on the front panel of the monitor. The function of the 3 keys during normal operation are summarized below:



**Figure 2-2 LCD Display and Keypad**

### Key Function in Normal Operation

**[MODE]** -Turn on/off the power\* and step through menu items

**[N/-]** -Toggle on/off the back light, negative acknowledge, decrease value

**[Y/+]** -Start measurement, positive acknowledge, increase value

\* Pressing and holding **[MODE]** key for 5 seconds turns off the power to the monitor. Monitor will beep once per second and display countdown timer during power-down sequence. Press **[MODE]** key momentarily to step through menu items. To save time, press any key during message scrolling to skip to the end of the message.

## 2.3 Power On/Off

**To turn on** the MiniRAE 2000 portable VOC monitor, press [MODE] key for one second and release. The audio buzzer will beep once and the air pump will turn on. The display will show “ON!..” and then “Ver n.nn” to indicate the unit’s current firmware version number. Next displayed are the serial number, the model number, Operating mode, current date and time, unit internal temperature, gas selected, high low, STEL, TWA/AVG alarm limits, battery voltage, and shut off voltage. Also displayed are internal mode settings such as User mode, Alarm mode, datalog time remaining and log periods in the respective order.

**To turn off** the MiniRAE 2000 portable VOC monitor, press and hold the [MODE] key for 5 seconds. The monitor will beep once per second during the power-down sequence with a count down timer showing the number of remaining seconds. The message “Off!..” flashes on the LCD display and the display will go blank indicating that the monitor is turned off.

### **Data protection during power off**

When the monitor is turned off, all the current real time data including last measured value are erased. However, the datalog data is preserved in non-volatile memory. Even if the battery is disconnected, the datalog data will not be lost. While the power is off, the real time clock will continue to operate until the battery is completely drained (usually in 4-5 days without any charging). If the battery is completely drained or is disconnected from the monitor for more than 30 minutes, the real time clock will be lost. In this case, the user needs to enter the real time clock information again, as described in Section 4, or send the PC clock during configuration through the PC communication.

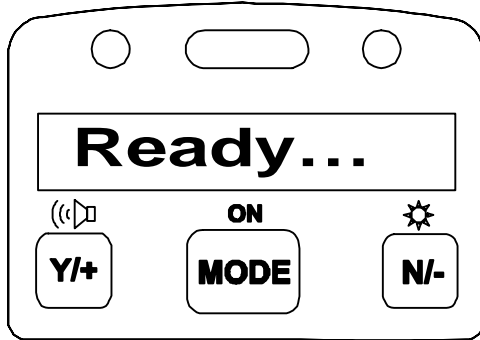


## 2.4 Operation

The **MiniRAE 2000** VOC monitor has two operation modes: **Survey** and **Hygiene** mode. The **Survey mode** allows the user to manually start and stop the monitoring/measuring operation and display certain exposure values. In the **Hygiene mode**, the monitor runs continuously after the monitor is turned on. Refer to Section 4.7.1 for switching between the two modes.

## 2.4.1 Survey Mode

After the monitor is turned on, it runs through the start up menu. Then the message “**Ready...**” is displayed (see figure below).



At this point, the user has two options:

1. Step through the Main Menu.
2. Take a measurement.

Press the [**MODE**] button to step through the Main Menu. Press the [**Y/+**] button to proceed to take a measurement.

### **The Main Menu**

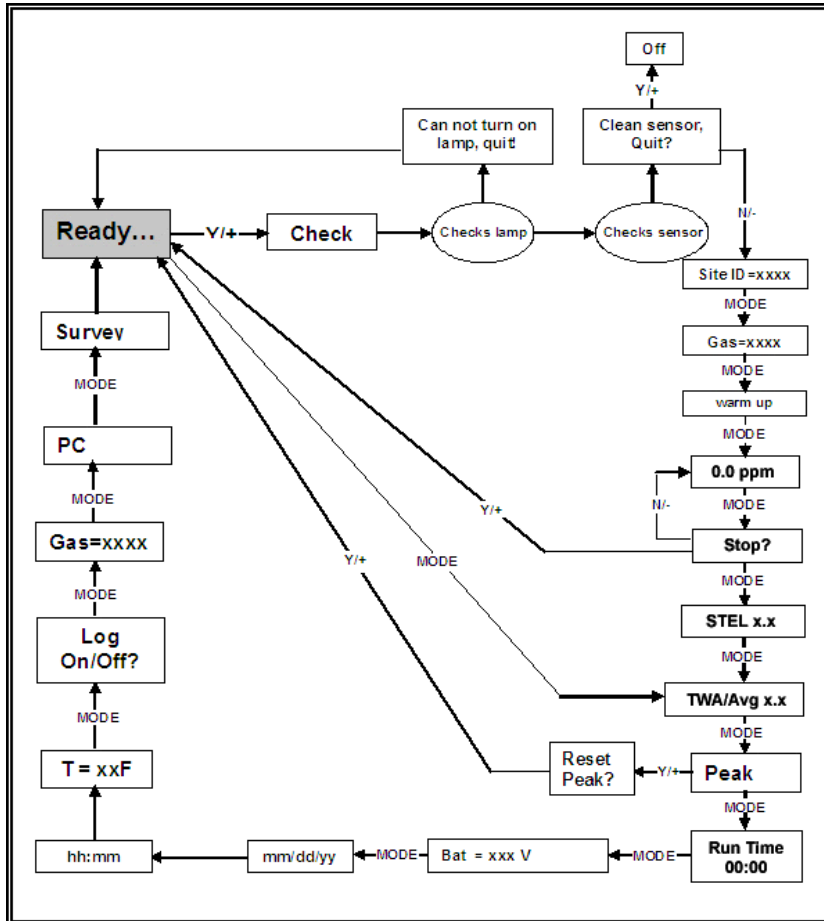
Press the **[MODE]** button to enter the Main Menu. Press the **[Y/+]**, **[MODE]** or **[N/-]** as indicated in the flow chart below to step through the Main Menu. The PID sensor and pump are turned off during this time.

The Main Menu functions are:

- **Ready...**
- **Check**
- **Site ID = xxxx**
- **Gas = xxxx**
- **Warm up**
- **X.x ppm**
- **Stop?**
- **TWA/Avg x.x ppm**
- **STEL x.x ppm**
- **Peak x.x ppm**
- **Run time hh:mm**
- **Bat = X.XV**
- **Mm/dd/yy**
- **hh:mm**
- **T = xxxF [date, time and temperature (°C or °F)]**
- **Log On/Off?**
- **PC Comm?**
- **Survey**

These functions are arranged in a “round robin” order. To select a specific function, press the button as shown below until the desired function appears.

## Main Menu



### **The Main Menu Functions**

- **Ready...:** Indicates that the monitor is ready to take a measurement or to step through the Main Menu. Press the [Y/+] button to advance to taking a measurement (read “Taking a Measurement” on Page 2-12 for details).

Note: The **Ready...** screen is skipped if the menu is cycled through while a measurement is running.

- **Check...:** This message displays while the system is checking the lamp and the sensor. If the lamp test succeeds, the system will progress to checking the sensor. If the lamp test does not succeed, the display will read **Can not turn on lamp, quit!**

- **Can not turn on lamp, quit!:** This message displays when the lamp does not turn on. The system will automatically return to **Ready...** allowing the user to test the lamp again. If the lamp fails a second time, turn the monitor off and refer to Section 7.2 “**PID Sensor & Lamp Cleaning / Replacement**”.

- **Clean Sensor, Quit?:** This message displays when the sensor requires cleaning. Press the [Y/+] button to turn the monitor off and clean the sensor. Press the [N/-] button and the system will progress to the **Site ID = xx**

- **Site ID = xxxx:** This display shows the Site ID and indicates that the monitor is about to start taking measurements (read “Taking a Measurement” for details)

Note: This display appears only after a measurement has been started. It does not appear when the user is cycling through the Main Menu and the monitor is idling.

- **Gas = xxxx:** This display identifies the gas to be measured and indicates that the monitor is about to take a measurement (read “Taking a Measurement” for details)

Note: This display appears only after a measurement has been started. It does not appear when the user is cycling through the Main Menu and the monitor is idling.

- **x.x ppm:** (read “Taking a Measurement” for details)

Note: This display appears only after a measurement has been started. It does not appear when the user is cycling through the Main Menu and the monitor is idling.

- **TWA/Avg:** Displays (in ppm) the Time Weighted Average (TWA) or the Average since the start of the measurement. The average is recalculated every minute.
- **STEL:** Displays the Short Term Exposure Limit.
- **PEAK:** Displays (in ppm) the highest instantaneous reading since the start of the measurement. If [Y/+] is pressed while the peak reading is displayed, the unit will ask **Reset Peak?**. If [Y/+] is pressed again, the peak value will be cleared and the display will return to the **Ready...** message or instantaneous reading. The peak reading is automatically reset when a new measurement is started by pressing [Y/+] from the **Ready...** screen.
- **Run time hh:mm:** The duration of the current measurement period.
- **Bat = X.XV:** The current battery voltage.

Note: A fully charged battery pack should show 4.8 volts or higher. When the battery voltage falls below 4.4 volts, a flashing “**Bat**” will appear as a warning message. At that point, you have 20-30 minutes of run time remaining. When the battery voltage falls below 4.2 volts the monitor turns off automatically.

- **Mm/dd/yy:** The current date.
- **hh:mm:** The current time (24-hour format)

- **T = xxxF:** The internal unit temperature in degrees Fahrenheit. (see Section 4.7.13 to change temperature units)
- **Log on/Off?:** Allows the user to start datalogging of the current measurement. A superscript “L” flashes in the ppm measurement display when datalogging is on. This screen is not shown when datalogging is disabled or when the monitor is not operating in manual start/stop mode.
- **PC Comm?:** This function enables the user to upload data from the MiniRAE 2000 to a Personal Computer (PC) or send/receive configuration information between a PC and the MiniRAE 2000. Press [MODE] to return to **Ready...** .

To communicate with a PC, connect the monitor to the serial port of a PC and start the MiniRAE 2000 application software. Press the [Y/+] button and the LCD displays “**pause monitor, ok?**” Press the [Y/+] button one more time, and the display shows “**Comm...**” The monitor is now ready to receive commands from the PC.

- **Survey:** This function displays the Current Operating Mode (**Survey** or **Hygiene**).

### **Taking a Measurement**

There are two ways to start a measurement.

1. Operating in Hygiene mode.
2. Manually start and stop measurement in Survey mode.

To start a measurement in Hygiene mode, please refer to Section 4.7.1, “**Change Operation Mode**”. To start a measurement in Survey Mode, the MiniRAE 2000 monitor must first be in the “Ready...” mode. This is the mode to which the monitor normally powers up.

### **Measurement phases**

1. Ready
2. Start measurement
3. Measurement display and datalogging
4. Stop measurement

#### **Ready**

The display reads **Ready...** indicating the unit is ready to start a measurement.

#### **Start Measurement**

Press the [Y/+] button to start the check cycle (see above), and then the measurement cycle.

After completing the **Check** cycle, the display will show the **Site ID** and then the **Gas** selected for measurement. The pump will start and the reading will be displayed. The **Peak** and **Average** values will be automatically reset to zero.

#### **Measurement Display and Datalog**

Instantaneous readings of the gas concentration in parts per million (ppm) are updated every second. A flashing superscript **L** is displayed when datalogging is on. Datalog information is saved only after one full datalog period is completed.



### **Stop Measurement**

Press the **[MODE]** button and the display shows Stop? Press **[N/-]** to continue measurement and **[Y/+]** to stop the measurement and datalog event. The pump stops automatically when measurement is stopped. Peak and average values for the current measurement can be read in idle mode until a new measurement is started.

### **Automatic Increment of Site ID**

Every time a measurement is taken, the site ID will be incremented by one automatically in Survey mode.

### **Variable Alarm Signal**

In Survey Mode, if the measurement exceeds the low alarm limit, the buzzer and flashing alarm are activated and will beep/flash once per second. The alarms will increase in frequency as the gas concentration approaches the high alarm limit reaching 8 times per second when the high alarm has been exceeded.

Press **[Y/+]** key to clear if latching alarm.

## 2.4.2 Hygiene Mode

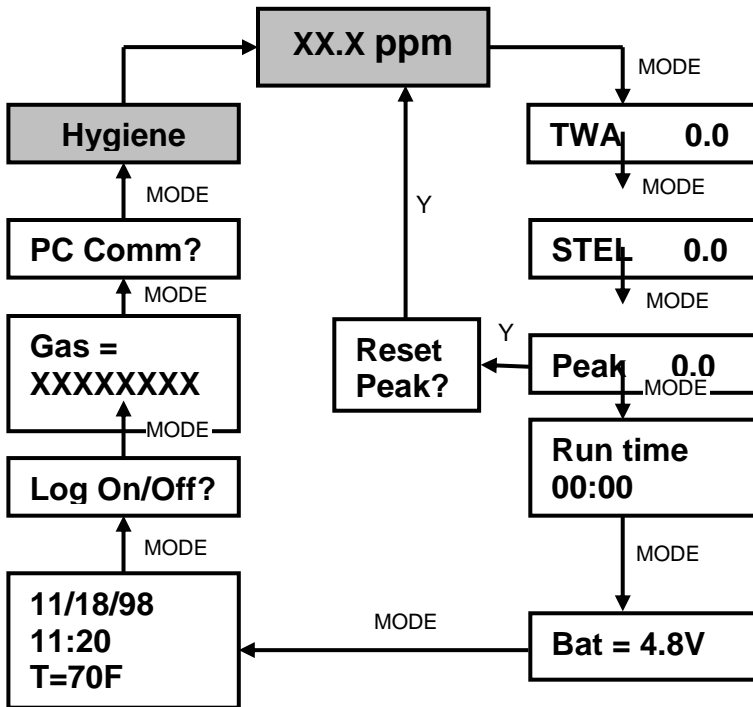
In Hygiene Mode, the unit will continuously taking measurements, once the power is turned on. After the initial start-up sequence displaying the current monitor settings, the LCD displays the instantaneous readings.

The Hygiene operation menu displays include:

- **Real time readings in ppm**
- **Current TWA/Avg, STEL and Peak values (see Section 4.6.6)**
- **Run time**
- **Current battery voltage**
- **Date, time and temperature**
- **Log on/off?**
- **Gas name**
- **PC communication?**
- **Hygiene**

Detailed description of most of these displays are the same as Section 2.4.1.

## HYGIENE MODE MAIN MENU



To choose a specific display, press the [MODE] key one or more times until the desired display appears, or the [Y/+] key where indicated with a Y.

**Note:** To get back to instantaneous reading from any of the above display, press [MODE] key repeatedly until the “XX.X ppm” display appears.

## **2.5 Alarm Signals**

During each measurement period, the gas concentration is compared with the programmed alarm limits (gas concentration alarm limit settings: Low, High, TWA and STEL). If the concentration exceeds any of the preset limits, the loud buzzer and red flashing LED are activated immediately to warn the user of the alarm condition.

In addition, the MiniRAE 2000 will alarm if one of the following conditions occurs: battery voltage falls below a pre-set voltage level (4.4 V), failure of UV lamp, pump stall, or when the datalog memory is full. When the low battery alarm occurs, there will be approximately 20-30 minutes of operating time remaining. When the battery voltage falls below 4.2 V, the monitor will turn off automatically.

### Alarm Signal Summary:

Condition	Alarm Signal
Gas exceeds “High Alarm” limit	3 beeps/flashes per second
Gas exceeds “Low Alarm” limit	2 beeps/flashes per second
Gas exceeds “TWA” limit	1 Beeps/flashes per seconds
Gas exceeds “STEL” limit	1 Beeps/flashes per seconds
Pump failure	3 beeps/flashes per second plus “Pump” message on LCD
PID lamp failure	3 beeps/flashes per second plus “Lamp” message on LCD
Low battery	1 flash per second, 1 beep per minute plus “Bat” message on LCD
Memory full	1 flash per second plus “Mem” message on LCD

### Alarm Signal Testing:

Under normal non-alarm conditions, it is possible to test the MiniRAE 2000 LED and buzzer in Special Diagnostic Mode (see Section 8 for details).

## 2.6 Preset Alarm Limits and Calibration

The MiniRAE 2000 portable VOC monitor is factory calibrated with standard calibration gas, and is programmed with default alarm limits. There are 102 gas settings stored in the library. Some examples of calibration and alarm limits are shown below. Refer to Section 4 on programming procedures for selecting a different gas, perform a calibration or set new alarm limits.

### Factory Calibration and Preset Alarm Limits

Cal Gas	Cal Span	unit	Low	High	TWA	STEL
Isobutylene	100	ppm	50	100	100	250
Hexane, n-	100	ppm	500	750	500	750
Xylene, m-	100	ppm	100	150	100	150
Benzene	5	ppm	2	5	5	2
Styrene	50	ppm	20	40	20	40
Toluene	100	ppm	50	100	50	100
Vinyl Chloride	10	ppm	5	10	5	10
Custom	100	ppm	50	100	50	100

## **2.7 Integrated Sampling Pump**

The MiniRAE 2000 portable VOC monitor includes an integrated sampling pump. This is a diaphragm type pump that provides a 500-600 cc per minute flow rate. Connecting a Teflon or metal tubing with 1/8 inch inside diameter to the gas inlet port of the MiniRAE 2000, this pump can pull in air samples from 200 feet away horizontally, or 90 feet vertically, at about 3 feet per second flow speed.

The pump turns on when a measurement is started, and turns off when the sample is manually stopped in Survey mode or when the unit is turned off from Hygiene Mode.

If liquid or other objects are pulled into the inlet port filter, the monitor will detect the obstruction and shut down the pump immediately. The alarm will be activated and a flashing error message “Pump” will be also displayed on the LCD display.

The user should acknowledge the pump shut off condition by clearing the obstruction and pressing the [Y/+] key to re-start the pump.

The pump stall threshold is set in the special Diagnostic Mode (Section 8).

## 2.8 Back Light

The LCD display is equipped with an LED back light to assist in reading the display under poor lighting conditions. Pressing and holding the [N/-] key for one second in normal operation can turn on the backlight. The backlight can be turned off by pressing [N/-] a second time. If the [N/-] key is not pressed, the back light will be turned off automatically after a pre-programmed time-out period to save power.

In addition, the ambient light is sensed and the back light will be turned on automatically if the ambient light is below a threshold level. The back light is turned off automatically when the ambient light exceeds the threshold level.

See Section 8 for instructions on how to set the light threshold level.

**Note:** The LED backlight consumes about 20-30% of the total average current, when the instrument is idle or not taking a measurement.



## **2.9 Datalogging**

During datalogging, the MiniRAE 2000 Portable VOC monitor flashes a superscript “L”, on the display to indicate that datalogging is enabled. The monitor stores the time stamp, sample number, and measured gas concentration at the end of every sample period (when data logging is enabled). In addition, the following information are stored: user ID, site ID, serial number, last calibration date, and alarm limits. All data are retained (even after the unit is turned off) in non-volatile memory so that it can be down loaded at a later time to a PC.

### **Datalogging event**

When Datalogging is enabled, measurement readings are being saved. These data are stored in “groups” or “events. A new event is created and stored each time the monitor is turned on, or a configuration parameter is changed, or datalogging is interrupted (e.g. Communication with PC during Hygiene mode). Information, such as start time, user ID, site ID, gas name, serial number, last calibration date, and alarm limits will be recorded.

### **Datalogging sample**

After an event is recorded, the unit records a shorter form of the data. This data contains: the sample number, time (hour/minute) and gas concentration.

### **3. OPERATION OF ACCESSORIES**

The accessories for the MiniRAE 2000 include:

- An AC Adapter (Battery Charger)
- Alkaline battery holder
- Water Trap Filter

Optional Accessories:

- Dilution Fitting
- Calibration adapter
- Calibration regulator and Flow controller
- Organic Vapor Zeroing kit

### 3.1 Standard Kit and Accessories

#### 1) AC Adapter (Battery Charger)

#### **WARNING**

**To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non-hazardous. Remove and replace battery only in area known to be non-hazardous.**

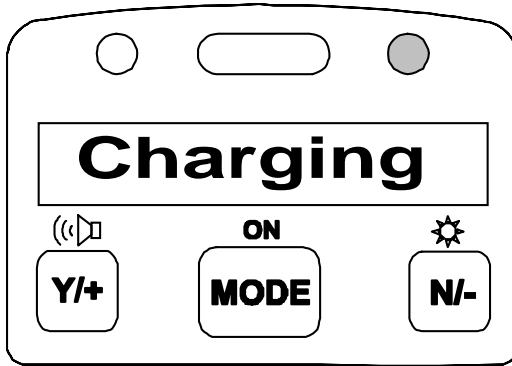
**Ne charger les batteries que dans emplacements designés non-dangereuses.**

A **battery charging circuit** is built into the MiniRAE 2000 monitor. It only needs a regular AC to 12 V DC adapter (wall mount transformer) to charge the monitor.

To charge the battery inside the MiniRAE 2000 monitor:

1. Power off the Monitor.
2. Connect the AC adapter (or the optional automotive charging adapter) to the DC jack on the MiniRAE 2000 monitor. If the unit was off, it will automatically turn on.
3. The first message displayed will be “Deep discharge?” The unit will ask this question for three times. If the user wants to discharge the battery pack, affirm this query with the [Y/+] key, otherwise the unit will move on to the charge mode directly.

4. While charging, the display message will alternate between “Charging” and “Bat=x.xV” (x.x is the present battery voltage). The LED should be red in color when charging.



5. When the battery is fully charged, the LED will change from red to green and the message “Fully charged” will appear on the display. After the battery is fully charged, the unit will enter the “trickle charge” mode. In which, the red LED will turn on for several seconds every minute, to maintain the full charge.

A completely discharged MiniRAE 2000 monitor will be charged to full capacity within 10 hours. The battery will be drained slowly even if the monitor is turned off. If the monitor has not been charged for 7-10 days, the battery voltage will be low.

The factory-supplied battery is designed to last for 10 hours of normal operation (no alarm, no back light condition), for a new battery under the best condition. As the battery becomes older or is subject to adverse conditions (such as cold ambient temperature), the battery capacity will be reduced significantly.

### **2) Alkaline Battery Holder**

An alkaline battery holder is supplied with each MiniRAE 2000. It accepts four AA size alkaline batteries and can be used in place of the Ni-MH or Ni-Cd battery pack to provide approximately 12-14 hours of operation. The adapter is intended to be used in emergency situations when there is no time to charge the Ni-Cd or Ni-MH battery pack.

To install the adapter, remove the cover of the battery compartment. Remove the Ni-Cd or Ni-MH battery pack from the battery compartment and replace with the alkaline battery adapter. Replace the battery compartment cover.

The internal charging circuit is designed to prevent damage to alkaline batteries and the charging circuit when alkaline batteries are installed inside the monitor.

**Note:** The AA Alkaline battery adapter supplied by RAE Systems Inc. is intrinsically safe!

### **3) Water Trap Filter**

The water trap filter is made of PTFE (Teflon<sup>®</sup>) membrane with a 0.45 micron pore size to prevent water from being sucked into the sensor manifold, which would cause extensive damage to the monitor. It will also remove any dust and other particles from entering the monitor and prolong the operating life of the sensor. To install the water trap, simply insert it to the front of the inlet tube of the MiniRAE 2000 monitor.

## 3.2 Optional Accessories

### 1) Dilution Fitting

The user may wish to install a dilution fitting on the inlet to dilute the gas samples. One application for a dilution fitting is to measure organic gas when the concentration exceeds the upper limit of the sensor range.

Make sure to set the dilution ratio in the programming mode (see Section 4.7.9) so that the correct gas reading will be displayed when the dilution fitting is used.

**WARNING: To use a dilution fitting, the user must have the monitor located in a clean atmosphere outside the confined space and use a remote access probe or Tygon tubing to measure the gas concentration inside the confined space.**

### 2) Calibration Adapter

The calibration adapter for the MiniRAE 2000 is a simple 6-inch Tygon tubing with a metal adapter on one end. During calibration, simply insert the metal adapter into the regular gas inlet probe of the MiniRAE 2000 and the tubing to the gas regulator on the gas bottle.

### **3) Calibration Regulator and Flow Controller**

The Calibration Regulator and Flow controller is used in the calibration process. It regulates the gas flow rate from the Span gas cylinder into the gas inlet of the MiniRAE 2000 monitor during calibration process. The maximum flow rate allowed by the flow controller is about 0.5L/min (500 cc per min.). Alternatively, a Demand-flow Regulator or a Tedlar gas bag may be used to match the pump flow precisely.

### **4) Organic Vapor Zeroing kit (Charcoal filter)**

The Organic Vapor Zeroing Kit is used for filtering organic air contaminants that may affect the zero calibration reading. To use the Organic Vapor Zeroing Kit, simply connect the filter to the inlet port of the MiniRAE 2000.

## **4. PROGRAMMING OF MINIRAE 2000**

The MiniRAE 2000 Monitor is built with a microcomputer to provide programming flexibility. Authorized users can recalibrate the monitor, change the alarm limits, change site ID, user ID, lamp type, and real time clock, etc.

Programming is menu-driven to provide intuitive end-user operation. The display shows the menu options and the key pad used for menu selection and data entry.



## 4.1 Programming Mode

The programming mode allows the users to change the setups in the monitor, calibrate the monitor, modify the sensor configuration and enter user information, etc. The programming mode has four menu items. Each menu item includes several sub-menus to perform additional programming functions. Appendix A shows a more detailed menu tree structure.

### Programming Menu

Calibrate/Select Gas?

Change Alarm Limits?

Change Datalog?

Change Monitor Setup?

Once inside the programming mode, the LCD will display the first menu. Each subsequent menu item can be viewed by pressing the [N/-] repeatedly until the desired menu is displayed. To enter the sub-menu of a particular menu, press [Y/+] key, the sub-menu will be displayed.

**Return to Operation mode:** To exit the programming mode and return to operation, press the [MODE] key once at any of the programming menu displays.

## 4.2 Keys for Programming Mode

The three keys perform a different set of functions during the programming mode as summarized below.

Key	Function in Programming Mode
[MODE]:	Exit menu when pressed momentarily or exit data entry mode when pressed and held for 1 second
[Y/+]:	Increase alphanumerical value for data entry or confirm (yes) for a question
[N/-]:	Decrease alphanumerical value for data entry or deny (no) for a question

### 4.3 Entering into Programming Mode

1. Turn on the MiniRAE 2000 monitor and wait for the **“Ready..”** message or the instantaneous reading display **“0.0 ppm”** message displayed.
2. Press and hold down both **[N/-]** and **[MODE]** keys for three seconds to enter programming mode. This delay is to prevent the user from entering programming mode by accident.
3. The first menu item **“Calibrate/select Gas?”** will be displayed.
4. Release both **[MODE]** and **[N/-]** keys simultaneously to start the programming mode
5. Press **[N/-]** key to scroll to the next menu item of the programming menu. Press **[Y/+]** key to select the displayed menu item.

The following Sections 4.4 - 4.7 describe the details of each menu options.

## 4.4 Calibrate and Select Gas

### WARNINGS

**The calibration of all newly purchased RAE Systems instruments should be tested by exposing the sensor(s) to known concentration calibration gas before the instrument is put into service for the first time.**

**For maximum safety, the accuracy of the MiniRAE 2000 should be checked by exposing it to known concentration calibration gas before each day's use.**

In the first menu of the programming mode, the user can perform functions such as calibration of the MiniRAE 2000 Monitor, select default cal memories, and modify cal memories (see Table 4.4).

Table 4.4

Calibrate/Select Gas Sub-Menu
Fresh Air Cal?
Span Cal?
Select Cal Memory?
Change Span Value?
Modify Cal Memory?
Change Correction Factor?

Calibrating the MiniRAE 2000 monitor is a two-point process using “fresh air “ and the standard reference gas (also known as span gas). First a “Fresh air” calibration, which contains no detectable VOC (0.0 ppm), is used to set the zero point for the sensor. Then a standard reference gas that contains a known concentration of a given gas is used to set the second point of reference.

**Note:** The span value must be set prior to calibrating for fresh air or span.

The user can store calibrations for up to 8 different measurement gases. The default gas selections are as follows:

Cal Memory #0.....Isobutylene

Cal Memory #1.....Hexane

Cal Memory #2.....Xylene

Cal Memory #3.....Benzene

Cal Memory #4.....Styrene

Cal Memory #5.....Toluene

Cal Memory #6.....Vinyl Chloride

Cal Memory #7.....Custom?

Memory #0 functions differently than the other 7 memories. For Memory #0, isobutylene is always the calibration gas. When the gas is changed in Memory #0 to one of 100 other preprogrammed chemicals or to a user-defined custom gas, a correction factor is applied to all the readings. During calibration, the unit requests isobutylene gas and displays the isobutylene concentration immediately following calibration, but when the unit is returned to the normal reading mode, it displays the selected gas and applies the correction factor.

The other 7 cal memories require the same calibration gas as the measurement gas. These memories may also be modified to a preprogrammed chemical or to a user-defined custom gas. In the gas library, only the gases that can be detected by the installed UV lamp will actually be displayed. Note that although the correction factor for the new gas will be displayed and can be modified, this factor is not applied when Memories #1-7 are

used. Therefore the factor will not affect the readings in these memories.

Once each of the memories has been calibrated, the user can switch between the calibrated gases by changing the cal memory without the need to recalibrate. Or the user can switch the measurement gas in Memory #0 and the appropriate correction factor will automatically be applied without the need to recalibrate. If the gas is changed in Memories #1-7, it is necessary to recalibrate.

To change a default gas from the list above to a library or custom gas, first go to Select Cal Memory (Section 4.4.3) and then proceed to Modify Cal Memory (Section 4.4.5) to enter the desired gas. If the desired compound does not appear in the preprogrammed library, the user can use the Custom\_VOC entry in the library, or the name and correction factor of any of the existing compounds can be changed as described in Section 4.4.5. A list of some 300 correction factors is given in Technical Note 106, available at the website [www.raesystems.com](http://www.raesystems.com).

### **4.4.1 Fresh Air Calibration**

This procedure determines the zero point of the sensor calibration curve. To perform a fresh air calibration, use the calibration adapter to connect the MiniRAE 2000 to a “fresh” air source such as from a cylinder or Tedlar bag (option accessory). The “fresh” air is clean dry air without any organic impurities. If such an air cylinder is not available, any clean ambient air without detectable contaminant or a charcoal filter can be used.

1. The first sub-menu shows: “Fresh air Cal?”
2. Make sure that the MiniRAE 2000 is connected to one of the “fresh” air sources described above.
3. Press the [Y/+] key, the display shows “zero in progress” followed by “wait..” and a countdown timer.

After about 15 seconds pause, the display will show the message “update data...zeroed... reading = X.X ppm...” Press any key or wait about 20 seconds, the monitor will return back to “Fresh air Calibration?” submenu.

### **4.4.2 Span Calibration**

This procedure determines the second point of the sensor calibration curve for the sensor. A cylinder of standard reference gas (span gas) fitted with a 500 cc/min. flow-limiting regulator or a flow-matching regulator is the simplest way to perform this procedure. Choose the 500 cc/min. regulator only if the flow rate matches or slightly exceeds the flow rate of the instrument pump. Alternatively, the span gas can first be filled into a Tedlar Bag, or delivered through a demand-flow regulator. Connect the calibration adapter to the inlet port of the MiniRAE 2000 Monitor, and connect the tubing to the regulator or Tedlar bag.

Another alternative is to use a regulator with >500 cc/min flow but allow the excess flow to escape through a T or an open tube. In the latter method, the span gas flows out through an open tube slightly wider than the probe, and the probe is inserted into the calibration tube.

Before executing a span calibration, make sure the span value has been set correctly (see next sub-menu).

1. Make sure the monitor is connected to one of the span gas sources described above.
2. Press the [Y/+] key at the “Span Cal?” to start the calibration. The display shows the gas name and the span value of the corresponding gas.
3. The display shows “Apply gas now!” Turn on the valve of the span gas supply.



4. Display shows “wait.... 30” with a count down timer showing the number of remaining seconds while the monitor performs the calibration.
5. To abort the calibration, press any key during the count down. The display shows “Aborted!” and return to “Span Cal?” sub-menu.
6. When the count down timer reaches 0, the display shows the calibrated value.

**Note:** The reading should be very close to the span gas value.

7. During calibration, the monitor waits for an increased signal before starting the countdown timer. If a minimal response is not obtained after 35 seconds, the monitor displays “No Gas!” Check the span gas valve is on and for lamp or sensor failure before trying again.
8. The calibration can be started manually by pressing any key while the “Apply gas now!” is displayed.
9. After a span calibration is completed, the display will show the message “Update Data Span Cal Done! Turn Off Gas.”
10. Turn off the flow of gas. Disconnect the calibration adapter or Tedlar bag from the MiniRAE 2000 Monitor.
11. Press any key and it returns back to “Span Gas Cal?”

### 4.4.3 Select Cal Memory

This function allows the user to select one of eight different memories for gas calibration and measurement. For Memories #1-7, the calibration and measurement gas is the same and no correction factor is applied. For Memory #0, the calibration gas is always isobutylene and the measurement gas may be different, in which case the correction factor for that gas is automatically applied. The default gas selections are listed in Section 4.4

1. “Select Cal Memory?” is the third sub-menu item in the Calibration sub-menu. Pressing the [Y/+] key, the display will show “Gas =” gas name followed by “Mem # x?”
2. Press [N/-] to scroll through all the memory numbers and the gas selections respectively. Press [Y/+] to accept the displayed Cal Memory number.
3. After the [Y/+] key is pressed, the display shows “Save?” Press [Y/+] key to save and proceed. Press [N/-] to discard the entry and advance to the next sub-menu.
4. If the gas in a newly selected Cal Memory number is not calibrated, the display shows “CF= x.xx”. A correction factor with the value “x.xx” will be applied.
5. If the gas of a newly selected cal memory number has been calibrated previously, the display shows “Last calibrated xx/xx/xx”.

#### 4.4.4 Change Span Value

This function allows the user to change the span values of the calibration gases.

1. “Change Span Value?” is the fourth sub-menu item in the Calibration sub-menu
2. Press [**Y/+**], display shows the gas name and the span value. A cursor will blink at the first digit of the Span value. To modify the span gas value, go to Step 3. Otherwise, press and hold the [**MODE**] key for 1 second to accept the previously stored span gas value and move to the next sub-menu.
3. Starting from the left-most digit of the span gas value, use the [**Y/+**] or [**N/-**] key to change the digit value and press [**MODE**] key momentarily to advance to next digit. Repeat this process until all digits are entered. Press and hold the [**MODE**] for 1 second to exit.
4. The display shows “Save?” To accept the new value, press the [**Y/+**] key. Press the [**N/-**] key or the [**MODE**] key to discard the change and move to the next sub-menu.

### 4.4.5 Modify Cal Memory

If the current cal memory number selected is not memory 0, users will be prompted whether to modify the settings of the selected cal memory. Press **[Y/+]** to modify the cal memory and **[N/-]** to go to the next sub-menu.

Once **[Y/+]** is pressed the LCD display will show the current memory number, current Gas selected and prompt user for acceptance of current gas selected.

1. Press **[N/-]** to modify the gas selection if desired. Or press **[Y/+]** key to skip the change of gas selection, and proceed to the next sub-menu.
2. After pressing **[N/-]**, display shows “Copy gas from library?” Press **[Y/+]** to accept or **[N/-]** for the next sub-menu, “Enter Custom gas?”
3. In the “Copy gas from library” submenu, use **[Y/+]** and **[N/-]** keys to scroll through the selections in the library. Press **[MODE]** key momentarily to select the gas. The display shows ”Save?” Press **[Y/+]** to save or **[N/-]** to discard the changes and proceed to next sub-menu.
4. In the Custom gas sub-menu, the user can enter the gas name. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all digits (up to 8 digits) of the custom gas name is entered.

Press and hold the **[MODE]** key for 1 second to exit the name entry mode. The display will show “Save?” Press **[Y/+]** to save the entry, or **[N/-]** to discard the changes.

### 4.4.6 Change Correction Factor

This function allows the user to change the Correction Factor of the standard calibration gas (only for Cal Memory #0).

1. “Change Correction Factor?” is the sixth sub-menu in the Calibration sub-menu.
2. Press **[Y/+]** key. Display shows the gas name, then the correction factor.

A cursor blinks at the left-most digit of the correction factor. If user wants to modify the correction factor, go to Step 3. Otherwise, press and hold the **[MODE]** key for 1 second to accept the previously stored correction factor value and return to the first sub-menu of the calibrate/select gas menu.

3. Starting from the left-most digit of the correction factor, use **[Y/+]** or **[N/-]** key to change the digit value and press **[MODE]** key momentarily to advance to the next digit, the cursor will move to the next digit to the right. Repeat this process until all digits are entered. Press and hold the **[MODE]** for 1 second to exit.
4. The display shows “Save?” To confirm the new value, press **[Y/+]** to accept the change. Press **[N/-]** or **[MODE]** to discard the change and return to the first sub-menu, Calibrate and Select Gas.

## 4.5 Change Alarm Limits

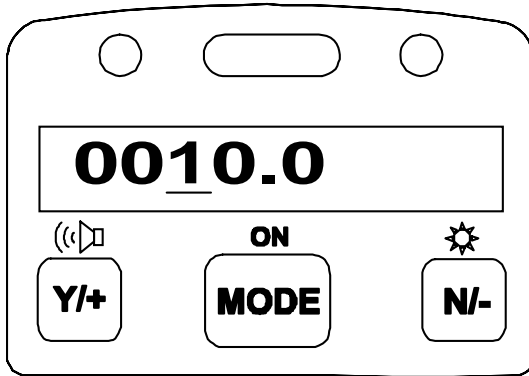
In this menu, the user can change the high and low alarm limits, the STEL limit and the TWA limit (see Table 4.5 below). Press the [Y/+] key and the display shows the current gas selected followed by the first sub-menu item below.

Table 4.5

<b>Alarm Limit Sub-Menu</b>
Change High Alarm limit?
Change Low Alarm limit?
Change STEL limit?
Change TWA limit?

1. Scroll through the Alarm Limit sub-menu using the [N/-] key until the display shows the desired limit to be changed, e.g., "High limit?", "STEL limit?", etc.

2. Press the **[Y/+]** key to select the desired limit and the display shows a flashing cursor on the left-most digit of the previously stored alarm limit.



3. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to its right. Repeat this process until the new limit value is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows “Save?” Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

### 4.5.1 Change Low Alarm Limit

The second sub-menu item in the Alarm Limit sub-menu allows the user to change the Low Alarm limit. The LCD displays “Low limit?” To change Low Alarm limit, press [Y/+] key, or Press [N/-] key advance to next sub-menu in Table 4.5.

1. Press [Y/+] and the display will show a flashing cursor on the left-most digit of the previously stored Low alarm limit.
2. To modify this limit value, use the [Y/+] or [N/-] key to change the digit value and press the [MODE] key momentarily to advance to the next digit. The flashing digit will move to the next digit to its right. Repeat this process until the new limit values is entered. Press and hold the [MODE] key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows “Save?” Press [Y/+] to accept the new value and move to the next sub-menu. Press [N/-] to discard the changes and move to the next sub-menu.



### 4.5.2 Change STEL Limit

This sub-menu item allows the user to change the STEL limit. The display shows “STEL limit?”

1. Press the **[Y/+]** key and the display will show a flashing cursor on the left-most digit of the previously stored STEL limit.
2. To modify this limit value, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new limit values is entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows “Save?” Press **[Y/+]** to accept the new value and move to the next sub-menu. Press **[N/-]** to discard the changes and move to the next sub-menu.

### 4.5.3 Change TWA Limit

This sub-menu item allows the user to change the TWA limit. The LCD displays “TWA limit?”

1. Press [**Y/+**] and the display will show a flashing cursor on the left-most digit of the previously stored TWA limit.
2. To modify this limit value, use the [**Y/+**] or [**N/-**] key to change the digit value and press the [**MODE**] key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new limit values is entered. Press and hold the [**MODE**] key for 1 second to exit data entry mode.
3. If there is any change to the existing value, the display shows “Save?” Press [**Y/+**] to accept the new value and move to the next sub-menu. Press [**N/-**] to discard the changes and move to the next sub-menu.

## **4.6 Change Datalog**

The MiniRAE 2000 monitor calculates and stores the concentration and ID of each sample taken. In the datalog sub-menu, a user can perform the tasks and functions shown below.

### **Datalog Sub-Menu**

Reset Peak/Minimum?

Clear Data?

Change Data Period?

Change Average Type?

### 4.6.1 Reset Peak

This function will reset the peak and minimum stored in the data memory. Note: this function will not clear the STEL or TWA data.

1. “Reset Peak?” is the first sub-menu item in the Datalog sub-menu (Table 4.6).
2. Press the **[Y/+]** key to reset the Peak/Minimum Values. The display shows “Are You Sure?”
3. Pressing the **[Y/+]** key again will reset the values. The display shows “Peak/Minimum Cleared” and moves to the next submenu.
4. Press the **[N/-]** or **[MODE]** key to exit without resetting the values and move to the next sub-menu.

### 4.6.2 Clear Data

This function will erase all data stored in the non-volatile datalog memory. Note: This function does not change STEL, TWA, Peak, Minimum and run time values, which are stored in the regular data memory.

1. “Clear Data?” is the third sub-menu item in the Datalog sub-menu.
2. Press the [Y/+] key to clear the datalog memory. The display shows “Are you sure?”
3. Press the [Y/+] key again to confirm erasure of all the datalog memory.
4. Press the [N/-] or [MODE] key to exit without clearing the datalog memory and move to the next datalog sub-menu.

### 4.6.3 Change Data Period

The datalog period can be programmed from 1 to 3,600 seconds (1 hour).

1. “Change Data Period?” is the fifth sub-menu item in the Datalog sub-menu.
2. Press the **[Y/+]** key and the display shows “Datalog Period = XXXX” with the left-most digit flashing, where “XXXX” is the previously stored data log period.
4. To modify this period, starting from the left-most digit, use the **[Y/+]** or **[N/-]** key to change the digit value and press the **[MODE]** key momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 4 digits of the new period are entered. Press and hold the **[MODE]** key for 1 second to exit data entry mode.
5. If there is any change to the existing value, the display will show “Save?” Press **[Y/+]** to accept the new value or **[N/-]** to discard the changes and move to the next sub-menu.

### 4.6.4 Change Average Type

The user can select either an 8-hour Time Weighted Average (TWA) or a running Average. The running average is simply the average of all instantaneous (1-second) readings since the measurement was started. This average may increase or decrease with time depending on the readings. The TWA is a cumulative value used to estimate the fraction of the 8-hour limit to which the user has been exposed since the start of the measurement. This value can only increase or remain constant, never decrease. Refer to Technical Note 119 for more information on how TWA is calculated.

1. “Change Average Type?” is the sixth sub-menu in the Datalog sub-menu.
2. Press the [Y/+] key to enter the function.
3. The display will show “Running Average?” or “Time Weighted Average?” depending on the current average type.
4. Press [N/-] key to toggle between the average types. Press [Y/+] key to select the displayed average type.
5. If there is any change to the existing setting, the display shows “Save?” Press [Y/+] to save the change. Press [N/-] or [MODE] to discard the change and return to the first sub-menu.

## 4.7 Change Monitor Setup

Several monitor specific variables can be changed in this menu. The following is a list of configuration data that can be modified by the user.

Monitor Setup Sub-Menu	Diagnostic Mode
Change Operation Mode?	“
Change Site ID?	Change Unit ID?
Change User ID?	Change Host ID?
Change Alarm Mode?	“
Change User Mode?	“
Change Date?	“
Change Time?	“
Change Lamp?	“
Change Pump Duty Cycle?	“
Change Unit?	“
Change Dilution Ratio?	“
Change Output?	“
Change DAC Range?	“
Set Temperature Unit?	“



### 4.7.1 Change Operation Mode

MiniRAE 2000 supports two operation modes: Survey and Hygiene mode.

**Survey mode:** Manual start/stop of measurements and display of certain exposure values.

**Hygiene mode:** Automatic measurements, running and datalogging continuously and calculates additional exposure values.

1. “Change Op Mode?” is the first sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the [Y/+] key and the display shows the current user mode: “Op Mode = *current mode*?”
3. Press the [Y/+] key to accept the currently displayed operation (Op) mode. Press [N/-] to toggle to the other operation mode. Press [MODE] to exit this sub-menu and move to the next monitor setup sub-menu.
4. When changing Op mode from Hygiene to Survey, the display shows the additional message “Warning! Exit Hygiene?” to prevent accidental exit from Hygiene mode. Press the [Y/+] key to acknowledge.
5. If there is any change to the existing setting, the display will show “Save?” Press the [Y/+] key to accept or the [N/-] key to discard and move to the next sub-menu.

**Note:** If a new Op Mode is saved, the display shows “Op Mode changed!!!” when exiting the programming mode.

### 4.7.2 Change Site ID

The user can enter an 8-digit alphanumeric site ID in the programming mode. This site ID will be included in the datalog report.

1. “Change Site ID?” is the second sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key and the display shows the current site ID: “Site ID = xxxxxxxx” with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 8 digits of the new site ID are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode.
5. If there is any change to the existing site ID, the display shows “Save?” Press the **[Y/+]** key to accept the new site ID. Press the **[N/-]** key to discard the change and move to the next sub-menu.

### 4.7.3 Change User ID

The user can enter an 8-digit alphanumeric user ID in the programming mode. This user ID will be included in the datalog report.

1. “Change User ID?” is the third sub-menu item the Monitor Setup menu.
2. Press the **[Y/+]** key and the display shows the current user ID: “User ID = xxxxxxxx” with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to cycle through all 26 letters and 10 numerals. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until all 8 digits of the new user ID are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode.
5. If there is any change to the existing user ID, the display shows “Save?” Press the **[Y/+]** key to accept the new user ID. Or press the **[N/-]** key to discard the changes and move to the next sub-menu.

### 4.7.4 Change Alarm Mode?

There are two different alarm modes: **Latched** and **Automatic Reset** (Auto Reset) in the MiniRAE 2000 that can be selected from the programming menu.

1. “Change Alarm Mode?” is the fourth sub-menu item in the Monitor Setup menu.
2. Press the [Y/+] key; the display shows the current alarm mode.
3. Press the [Y/+] key to accept the currently displayed alarm mode. Press [N/-] key to toggle to the other alarm mode. Press [MODE] to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing setting, the display will show “Save?” Press [Y/+] to save the change. Press [N/-] or [MODE] to discard the change and move to the next sub-menu.

### 4.7.5 Change User Mode

There are two different user modes: **Display** and **Program** that can be selected from the programming menu.

1. “Change User Mode?” is the fifth sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the [Y/+] key; the display shows the current user mode selected.
3. Press the [Y/+] key to accept the currently displayed user mode. Press [N/-] key to toggle to the alternate user modes. Press [MODE] to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing selection, the display shows messages “Program change” and “Are you sure?” Press [Y/+] to confirm the change or press [N/-] or [MODE] to discard the changes and move to the next sub-menu.

**CAUTION:** If the user mode is changed to **Display** mode, the user can no longer enter the programming mode. Therefore, the user can not change the user mode back to **Program** mode in normal mode.

To restore the user mode back to **Program** mode, turn the unit off and back on in Diagnostic Mode. Next enter Program mode by holding the [MODE] and [N/-] keys for three seconds. Enter the password at the prompt (the default is 0000). Once program mode is entered, go to the “Change Monitor Setup” / “Change User Mode” and change the mode back to **Program**.

An alternative way to change Display mode back to Program mode is through the PC and the ProRAE-Suite software.

### 4.7.6 Change Date

The MiniRAE 2000 monitor is equipped with a real time clock (RTC). The user can enter the correct date and time (see 4.7.7) for the real time clock.

1. “Change Date?” is the sixth sub-menu item in the Monitor Setup menu.
2. Press [**Y/+**] and the display shows the current date “mm / dd / yy” with the left most digit of the date flashing.
5. To modify this value, use the [**Y/+**] or [**N/-**] key to change the digit value and press the [**MODE**] key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new date and time values are entered. Press and hold the [**MODE**] key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows “Save?” Press [**Y/+**] to confirm the new value or press [**N/-**] or [**MODE**] to discard the changes and move to the next sub-menu.

### 4.7.7 Change Time

To change the time in the RTC of the MiniRAE 2000:

1. “Change Time?” is the seventh sub-menu item in the Monitor Setup menu.
2. Press [**Y/+**] and the display shows the current time in the 24-hour format “hh : mm” with the left most digit of the time flashing.
3. To modify this value, use the [**Y/+**] or [**N/-**] key to change the digit value and press the [**MODE**] key momentarily to advance to the next digit. The flashing digit will move on to next digit to its right. Repeat this process until the new date and time values are entered. Press and hold the [**MODE**] key for 1 second to exit data entry mode.
4. If there is any change to the existing value, the display shows “Save?” Press [**Y/+**] to confirm the new value or press [**N/-**] or [**MODE**] to discard the changes and move to the next sub-menu.

### 4.7.8 Change Lamp

There are three UV lamps with different photon energies available for the PID sensor: **9.8 eV**, **10.6 eV** and **11.7 eV**. The user can select any one of the lamps from the programming mode.

1. “Change Lamp Type?” is the eighth sub-menu item in the Monitor Setup menu (Table 4.7).
2. Press the **[Y/+]** key; the display shows the current PID lamp selection.
3. Press the **[Y/+]** key to accept the currently displayed lamp. Press **[N/-]** key to scroll through the sub-menu for other lamp selections. Press **[MODE]** to exit this sub-menu and return to the next sub-menu in Table 4.7.
4. If there is any change to the existing selection, the display will show “Save?” Press **[Y/+]** to save the new selection or press **[N/-]** or **[MODE]** to discard the change and return to the next sub-menu in Table 4.7.



### 4.7.9 Change Unit

User can change the display and datalog unit from parts per million (ppm) to milli-gram per cubic meter ( $\text{mg}/\text{m}^3$ ).

1. “Change Unit?” is the ninth sub-menu item in the Monitor Setup sub-menu.
2. Press the [Y/+] key, the display should show the current unit “Display Unit = ppm?” or “Display Unit =  $\text{mg}/\text{m}^3$ ?”
3. Press [Y/+] key to accept the currently displayed unit. Press [N/-] key to toggle to the other unit. Press [MODE] key to exit this sub-menu.
4. If there is any change to the existing selection, press [Y/+] to confirm the new selection or press [N/-] or [MODE] to discard the changes and move to the next sub-menu.

#### **Caution:**

1. **The correction factor in the gas library is calculated based on “ppm” unit. If “mg” unit is selected, the built-in correction factor library is not valid.**
2. **No automatic conversion between “ppm” and “ $\text{mg}/\text{m}^3$ ” reading is performed by the monitor.**
3. **When the unit name is changed from “ppm” to “mg”, the unit must be recalibrated with the span gas concentration entered in  $\text{mg}/\text{m}^3$ . The converse rule applies when the unit is changed from “mg” to “ppm”.**

### 4.7.10 Change Dilution Ratio

If a dilution system is used upstream of the MiniRAE 2000 inlet port, the user can enter the dilution ratio (from 1 to 10) to compensate the readings. The unit will then display the actual concentration of the gas before dilution. The dilution ratio should be 1 in normal operation where no dilution gas is applied to the sample gas. Dilution improves accuracy and linearity when the concentrations are above a few thousand ppm.

1. “Change Dilution Ratio?” is the tenth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key; the display shows the current dilution ratio: “Dilution Ratio = xx” with the left most digit flashing.
3. Press the **[Y/+]** or **[N/-]** key to increase or decrease the value of the digit. Press **[MODE]** momentarily to advance to the next digit. The flashing digit will move to the next digit to the right. Repeat this process until both digits of the new dilution ratio are entered.
4. Press and hold the **[MODE]** key for 1 second to exit the data entry mode and move to the next sub-menu.
5. If there is any change to the existing dilution ratio, the display shows “Save?” Press **[Y/+]** to confirm the new value or press **[N/-]** or **[MODE]** to discard the changes and move to the next sub-menu.

### 4.7.11 Change Output?

There are two different external output options: DAC (Analog output) and Alarm in the MiniRAE 2000 that can be selected from the programming menu. The alarm output can be used to connect to the optional vibration alarm (vibrator) only. The analog output, which is proportional to the gas concentration, can be connected a chart recorder or can be queried by a computer to download data in real time (see Technical Note 141).

1. “Change External Output?” is the eleventh sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key and the display shows the current output option selection: “Output = DAC?”
3. Press the **[Y/+]** key to accept the currently displayed output option. Press **[N/-]** to change to the other external option: “Output = Alarm?” Press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.
4. If there is any change to the existing selection the display will show “Save?” Then, press **[Y/+]** to save the change, press **[N/-]** to go back to Step 2, or press **[MODE]** to exit this sub-menu and move to the next monitor setup sub-menu.

### 4.7.12 Change DAC Range?

There are four different DAC (Digital-to-Analog Conversion) range values available in the **MiniRAE 2000: 20, 200, 2000 and 10K ppm**. The maximum 2.5V DC analog signal output from the unit will represent the range value chosen. (See for analog signal output connection.)

1. “Change DAC Range?” is the twelfth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key, the display shows the current DAC Range value: “DAC Range = 2000 ppm?”
3. Press the **[Y/+]** key to accept the currently displayed value. Press **[N/-]** to scroll through the sub-menu for other range values. Press **[MODE]** to exit this sub-menu and return to the first sub-menu in Table 4.7.
4. If there is any change to the existing selection, press the **[Y/+]** key and the display will show “Save?” Press the **[Y/+]** key to save the change or press the **[N/-]** key to discard and return to the first sub-menu in Table 4.7.

### 4.7.13 Set Temperature Unit?

The temperature display can be switched between Fahrenheit and Celsius units.

1. “Set Temperature Unit?” is the thirteenth sub-menu item in the Monitor Setup menu.
2. Press the **[Y/+]** key, and the display shows the current setting: “Temperature Unit = Fahrenheit?”
3. Press the **[Y/+]** key to accept the currently displayed value. Press **[N/-]** to select the sub-menu “Temperature Unit = Celsius?” Press **[MODE]** to exit this sub-menu and return to the first sub-menu in Table 4.7.
4. If there is any change to the existing selection, press the **[Y/+]** key and the display will show “Save?” Press the **[Y/+]** key to save the change and return to the first sub-menu in Table 4.7 or press the **[N/-]** key to discard and return to Step 3..

## 4.8 Exit Programming Mode

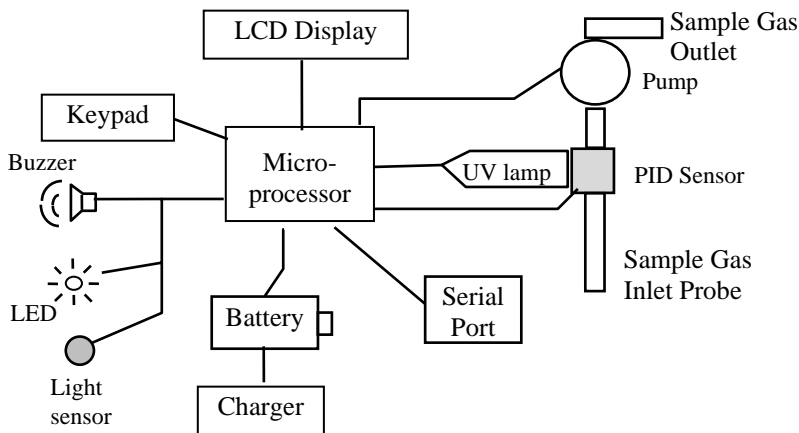
1. To exit programming mode from the first tier menu level, press the **[MODE]** key once.
2. To exit programming mode from 2nd tier sub-menu, press the **[MODE]** key twice.
3. To return to programming mode, press and hold down both the **[MODE]** and **[N/-]** keys for 3 seconds.

## 5. THEORY OF OPERATION

The MiniRAE 2000 monitor uses a newly developed electrodeless discharge UV lamp as the high-energy photon source for the PID. As organic vapors pass by the lamp, they are photo-ionized and the ejected electrons are detected as a current. The PID sensor with a standard 10.6 eV lamp detects a broad range of organic vapors. A lamp with high photon energy (e.g. 11.7 eV) will measure the more kinds of compounds, whereas low photon energies (e.g. 9.8 eV) are selective for easily ionizable compounds such as aromatics. In principle, any compound with an ionization energy lower than that of the lamp photons can be measured.

The PID sensor for the MiniRAE 2000 monitor is constructed as a small cavity in front of the UV lamp. A diaphragm pump draws the gas sample into the sensor and then pumps it out through the side of the instrument.

**Figure 5-1 MiniRAE 2000 Block Diagram**



A single chip microcomputer is used to control the operation of the alarm buzzer, LED, pump and light sensor. It measures the sensor readings and calculates the gas concentrations based on calibration to known standard gases. The data are stored in non-volatile memory so that they can be sent to a PC for record keeping. RS-232 transceivers provide a serial interface between the monitor and the serial port of a PC. An LCD display consisting of a single row of eight alpha/numeric characters is used to display the readings. The user interacts with the monitor through three keys on the front panel keypad.

A rechargeable NiMH, NiCd battery, or an alkaline battery pack powers the monitor.



## 6. MAINTENANCE

The major maintenance items of the MiniRAE 2000 are:

- Battery pack
- Sensor module
- PID lamp
- Sampling pump
- Inlet connectors and filters

**Note: Maintenance should be performed by qualified personnel only.**

**NOTE: The printed circuit board of the monitor is connected to the battery pack even if the power is turned off. Therefore, it is very important to disconnect the battery pack before servicing or replacing any components inside the monitor. Severe damage to the printed circuit board or battery may occur if the battery pack is not disconnected before servicing the unit.**

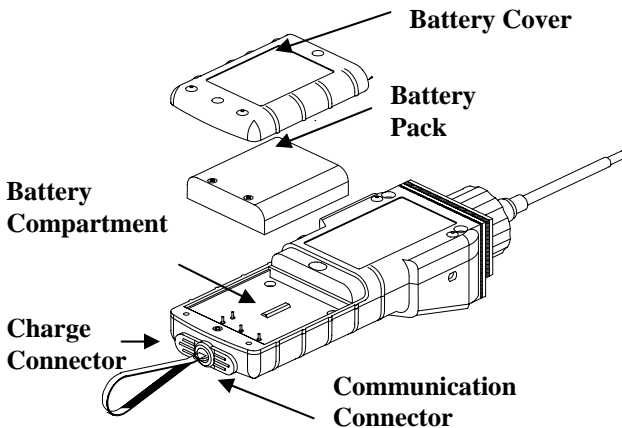
## 6.1 Battery Charging and Replacement

When the display shows a flashing message “Bat”, the battery requires recharging (see Section 3.1 for Battery charging). It is recommended to recharge the MiniRAE 2000 monitor upon returning from fieldwork. A fully charged battery runs a MiniRAE 2000 monitor for 10 hours continuously. The charging time is less than 10 hours for a fully discharged battery. The built-in charging circuit is controlled by the micro-controller to prevent over-charging. The battery may be replaced in the field (in area known to be non-hazardous) if required.

### WARNING

**To reduce the risk of ignition of hazardous atmospheres, recharge battery only in area known to be non- hazardous. Remove and replace battery only in area known to be non-hazardous.**

### Replacing Battery Pack



**Figure 6-1 Battery Replacement**

1.

Turn off the power of the MiniRAE 2000.

2. Unscrew the two battery compartment screws, located on the bottom of the monitor, and remove the cover.
3. Remove the battery pack from the battery compartment.
4. Replace a fully charged spare battery pack inside the battery compartment. Make sure the battery pack is oriented properly inside the compartment
5. Close the battery cover and tighten the two screws.

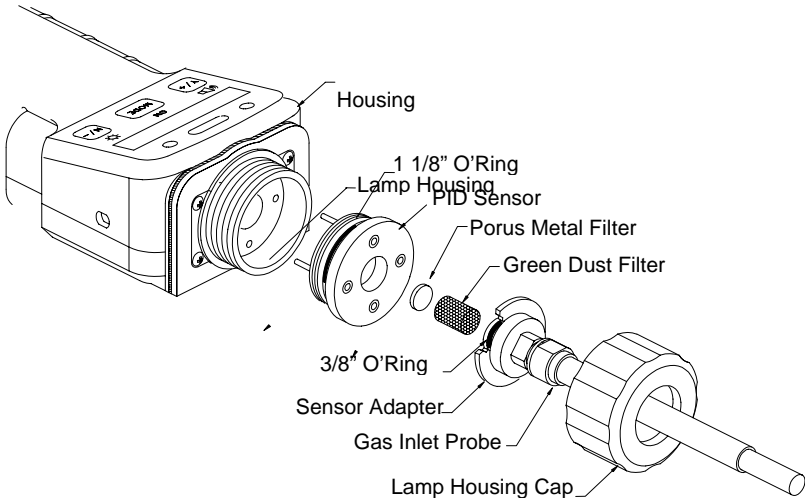
### **Replacing Alkaline Battery Adapter**

1. Insert four fresh AA size alkaline batteries into the alkaline battery holder. Make sure that the polarity of the batteries is correct.
2. Follow the same procedure as described above to replace the battery holder.

**Note:** The internal charging circuit is designed to prevent charging to alkaline batteries.

## 6.2 PID Sensor & Lamp Cleaning/Replacement

The sensor module is made of several components and is attached to the lamp-housing unit as shown in Figure 7-2.



**Figure 7-2 Sensor Components**

**Note:** Normally the cleaning procedure is not needed. Clean the PID sensor module, the lamp and the lamp housing only when one of the following happened:

1. The reading is inaccurate even after calibration.
2. The reading is very sensitive to air moisture.
3. A chemical liquid has been sucked into the unit and damaged the unit.

Use of the water trap filter will help prevent contamination and accidentally drawing liquid into the sensor.

To access the sensor components and lamp, gently unscrew the lamp-housing cap, remove the sensor adapter with the gas inlet probe and the metal filter all together. Then hold the PID sensor and pull straight out to avoid bending the electrical pins on the sensor (see Figure 7-2). A slight, gentle rocking motion helps release the sensor.

### **To clean the PID sensor:**

Place the entire PID sensor module into GC grade methanol. It is highly recommended that an ultrasound bath to be used to clean the sensor for at least 15 minutes. Then dry the sensor thoroughly. Never touch the electrodes of the sensor by hand.

Also use a methanol-soaked cotton swab to wipe off the lamp housing where it contacts the sensor when the sensor is installed.

Turn over the sensor so that the pins point up and the sensor cavity is visible. Examine the sensor electrodes for any corrosion, damage, or bending out of alignment. The metal sensor electrode “fingers” should be flat and straight. If necessary, carefully bend the sensor fingers to ensure that they do not touch the Teflon portions and that they are parallel to each other. Make sure that the nuts on the sensor pins are snug but not overtight. If the sensor is corroded or otherwise damaged, it should be replaced.

### **To clean lamp housing or change the lamp:**

### **To clean lamp housing or change the lamp:**

If the lamp does not turn on, the monitor will display an error message to indicate replacement of the lamp may be required.

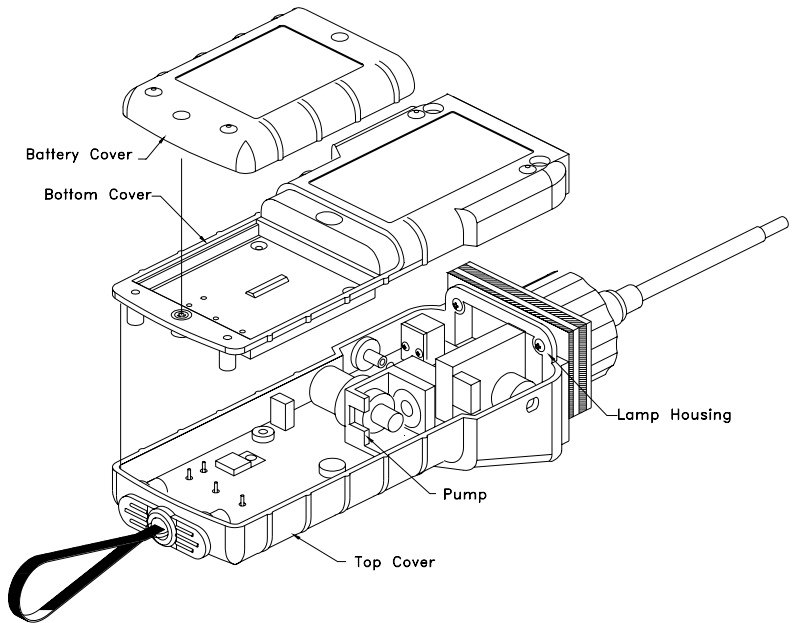
1. If the lamp is operational, clean the lamp window surface and the lamp housing by wiping it with GC grade methanol using a cotton swab using moderate pressure. After cleaning, hold the lamp up to the light at an angle to detect any remaining film. Repeat the process until the lamp window is clean. Never use water solutions to clean the lamp. Dry the lamp and the lamp housing thoroughly after cleaning.

**CAUTION: Never touch the window surface with the fingers or anything else that may leave a film. Never use acetone or aqueous solutions.**

2. If the lamp does not turn on, remove the lamp from the lamp housing. Place the lamp O-ring onto the new lamp. Insert the new lamp, avoiding contact with the flat window surface.
3. Reinstall the PID sensor module.
4. Tighten the Lamp Housing Cap.
5. If the lamp type has been changed, adjust the lamp type setting in the programming mode (Section 4.7.8).

## 6.3 Sampling Pump

When approaching the end of the specified lifetime of the pump, it will consume higher amount of energy and reduce its sample draw capability significantly. When this occurs, it is necessary to replace or rebuild the pump. When checking the pump flow, make sure that the inlet connector is tight and the inlet tubing is in good condition. Connect a flow meter to the gas inlet probe. The flow rate should be above 450 cc/min when there is no air leakage.



**Figure 7-3 Sampling Pump**

### **Pump Replacement**

1. Turn off the MiniRAE 2000 power.
2. Open the battery cover, remove the battery pack, and carefully unscrew the six screws to open the bottom cover.
3. Unplug the pump from the PCB. Unscrew the two screws that hold the pump assembly to the PCB. Disconnect the Tygon tubing that connects the pump to the gas inlet port and gas outlet port.
4. Insert a new pump assembly. Connect the Tygon tubing to the gas inlet port. Plug the pump connector back into the PCB and screw down the pump assembly to the board.
5. Replace the bottom cover and tighten the six screws. Reconnect the battery pack. Replace the battery pack and its cover.



## 6.4 Turning on the UV Lamp

The UV lamp is made of a glass envelope and a UV window (salt crystal) on one end of the envelope. The inside of the lamp is filled with low pressure gases. To turn on the lamp, a high voltage electric field is applied from the outside of the glass envelope. The molecules inside the lamp are ionized and produce a glow discharge that generates the UV light. The MiniRAE 2000 has a built-in sensing mechanism to monitor the status of the UV lamp and display a “Lamp” error message if it is not on.

If the UV lamp has not been used for a long period of time ( $> 1$  month) or is cold, it may become slightly harder to turn on. If such a condition occurs, the “Lamp” message will appear in the monitor display during the power on sequence. This phenomenon is more significant in 0.25” UV lamps used in ToxiRAE and MultiRAE Plus products, because of the relatively small lamp size. To solve this problem, simply turn on and off the monitor a few times and the lamp should turn on. After the UV lamp is turned on for the first time, it should be easier to turn on the UV lamp next time.

It is possible that the UV lamp is actually on when the lamp error message appears. This is because when the lamp becomes old, the internal threshold level to detect lamp failure may have shifted and cause a false alarm. To eliminate such possibility, simply check to see the UV lamp is actually on. This can be done easily by removing the sensor cap and observing the glow light of the UV lamp in a dark place. The user can also feed the monitor with calibration gas and observe if the sensor reading changes. If the reading changes significantly with the gas, the UV lamp is actually on.

A possible failure mechanism for the UV lamp is a leak developed along the seal of the glass envelope. When such condition occurs, the lamp will become very hard or impossible to turn on and will need to be replaced.

## 7. TROUBLESHOOTING

To aid the user in diagnosing the monitor, a special diagnostic mode can be used displays critical, low level parameters. Section 7.1 describes the operation of the diagnostic mode. Section 7.2 summarizes the frequently encountered problems and suggested solutions. By turning on the MiniRAE 2000 monitor in diagnostic mode and by using the troubleshooting table in Section 7.2, the user can usually correct the problem without having to return the monitor for repair.

### **WARNING**

**This function should be used by qualified personnel only! The diagnostic mode allows the user to set several low-level parameters that are very critical to the operation of the monitor. Extra care should be taken when setting these parameters. If the user is not familiar with the function of these parameters and sets them incorrectly, it may cause the monitor to shut down or malfunction.**

## 7.1 Troubleshooting Table

Problem	Possible Reasons & Solutions
Cannot turn on power after charging the battery	<p><b>Reasons:</b> Discharged battery. Defective battery. Microcomputer hang-up.</p> <p><b>Solutions:</b> Charge or replace battery. Disconnect, then connect battery to reset computer.</p>
No LCD back light	<p><b>Reasons:</b> Trigger level too low, the current mode is not user mode, and the mode does not support automatic turn on back light.</p> <p><b>Solutions:</b> Adjust trigger level. Verify the back light can be turned on in user mode. Call authorized service center.</p>
Lost password	<p><b>Solutions:</b> Call Technical Support at +1.408 .752 .0723 or +1. 888 .723 .4800</p>
Reading abnormally High	<p><b>Reasons:</b> Dirty sensor module. Dirty water trap filter. Excessive moisture and water condensation.</p> <p><b>Solutions:</b> Clean sensor module and lamp housing. Replace water trap filter. Blow dry the sensor module.</p>
Buzzer Inoperative	<p><b>Reasons:</b> Bad buzzer.</p> <p><b>Solutions:</b> Call authorized service center.</p>

## TROUBLESHOOTING

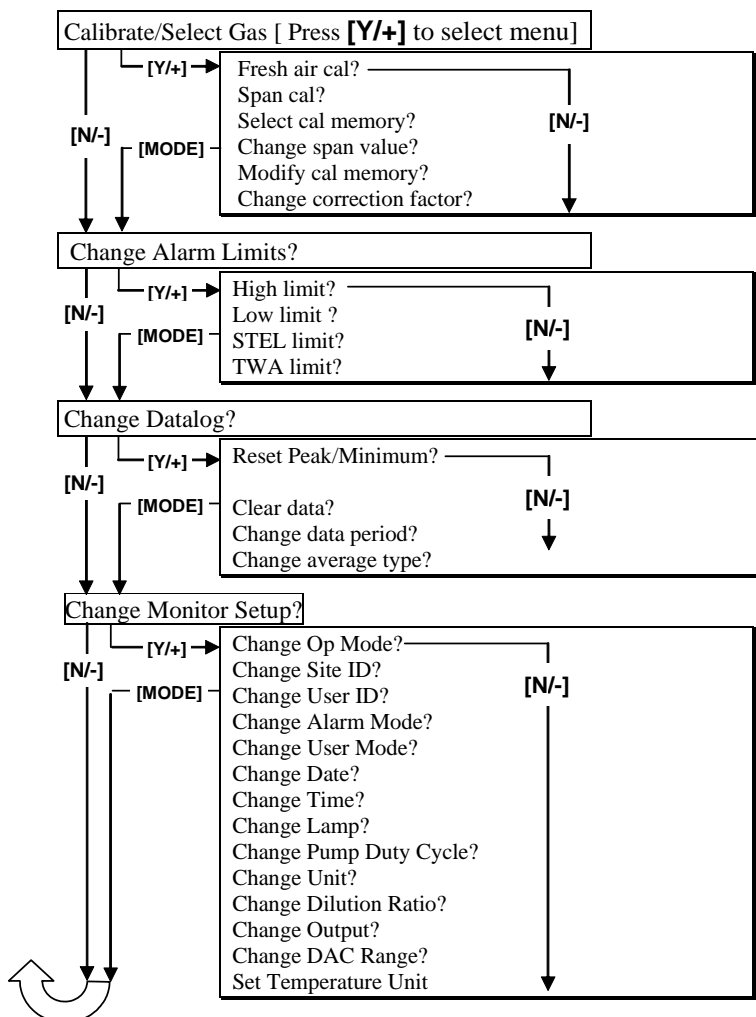
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Inlet flow too low	<p><b>Reasons:</b> Pump diaphragm damaged or has debris. Flow path leaks.</p> <p><b>Solutions:</b> Check flow path for leaks; sensor module O-ring, tube connectors, Teflon tube compression fitting. Replace pump or diaphragm.</p>
“Lamp” message during operation	<p><b>Reasons:</b> Lamp drive circuit. Weak or defective PID lamp, defective.</p> <p><b>Solutions:</b> Turn the unit off and back on Replace UV lamp</p>
Full scale measurement in humid environment	<p><b>Reasons:</b> Dirty or wet sensor.</p> <p><b>Solutions:</b> Clean and dry sensor and lamp housing. Adjust sensor fingers to ensure not touching Teflon. Use water trap filter.</p>
Reading abnormally low	<p><b>Reasons:</b> Incorrect calibration. Low sensitivity to the specific gas. Weak or dirty lamp. Air leakage.</p> <p><b>Solutions:</b> Calibrate the monitor. Replace sensor. Clean or replace lamp. Check air leakage.</p>

## APPENDIX A. QUICK REFERENCE GUIDE

Press [N/-] and [MODE], simultaneously, for 3 seconds, to enter Programming Mode. Press [MODE] to return to Survey Mode.

### PROGRAMMING MODE





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## RAE Systems, Inc. Contact Information

**Main Office:** 3775 N. First St.  
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**Telephone:** 408-952-8200

**Fax:** 408-952-8480

**Instrument Sales:** 877-723-2878

**Email:** [RaeSales@raesystems.com](mailto:RaeSales@raesystems.com)

**Website:** [www.raesystems.com](http://www.raesystems.com)

**Technical Service:** 888-723-4800  
[Tech@raesystems.com](mailto:Tech@raesystems.com)

### Special Note

If the monitor needs to be serviced, contact either:

1. The RAE Systems distributor from whom the monitor was purchased; they will return the monitor on your behalf.
2. The RAE Systems Technical Service Department. Before returning the monitor for service or repair, obtain a Returned Material Authorization (RMA) number for proper tracking of your equipment. This number needs to be on all documentation and posted on the outside of the box in which the monitor is returned for service or upgrade. Packages without RMA Numbers will be refused at the factory.

# Water Level Indicator with Laser-Marked Cable

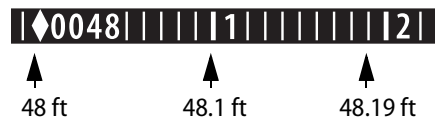


## Taking Readings

1. Switch on. Set sensitivity to 5 or 6.
2. Lower probe into well. When probe touches water, light turns on and beeper sounds.
3. Read depth to water from cable mark that aligns with your reference, such as the top of the well. Then switch off.

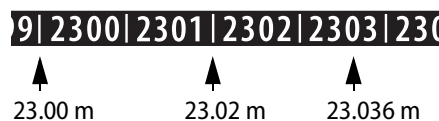
## English-Unit Cables

Divisions are 0.01 foot. Numerals mark feet and tenths of feet.



## Metric-Unit Cables

Centimeters are numbered. Numerals also serve as 2 mm graduations. Centimeter marks are crossed with 1, 2, or 3 lines at 100, 200, and 300 m.



## Indicator Controls

**On/Off/Sensitivity:** The on/off switch also adjusts sensitivity. Sensitivity is set properly if beeper and light turn off immediately when probe is removed from contact with water. Use lower setting for very conductive water or to eliminate false triggering. Use higher setting for less conductive water.

**Battery Test Button:** Push the button to check the batteries. Light and beeper activate when batteries are good.

## Replacing Batteries

The indicator uses two AA-size batteries. Press Test button to check batteries. Replace batteries if light and beeper do not activate.

1. Use coin or screwdriver to open battery holder. (  $\frac{1}{4}$  turn counter-clockwise )
2. Remove the two AA batteries. Insert new batteries with + terminals up, toward the cap.
3. Replace cap.

## Cleaning the Indicator

**Probe:** Wash probe with detergent.

**Reel:** Wipe off the reel with a damp cloth. Do not immerse in water.

**Cable:** Wash the cable with a laboratory-grade detergent such as Alconox or Liquinox. Rinse with distilled water as required. Remove oily deposits with dish-washing detergent. Do not leave the cable immersed in detergent for a long time. Rinse in distilled water.

Do not use nitric acid, hydrochloric acid, MEK, Acetone, Toluene, or alcohol to clean the cable. Even short-term exposure to these substances can damage the polyurethane cable jacket.

## Repairs

If your water level indicator is damaged, you can order replacement parts to fix it yourself or you can return it to the factory for repair.

To order replacement parts, please visit [www.slopeindicator.com](http://www.slopeindicator.com). Click on the link for Support, and then click on Water Level Indicator Parts.

To return the water level indicator to the factory for repair, contact the Slope Indicator factory for a Return Authorization:

Tel: 425-493-6200

Fax: 425- 493-6250

Email: [solutions@slope.com](mailto:solutions@slope.com)

The factory will email or fax you the return authorization. Check that the indicator is clean and dry and package it. Write the return authorization number on the outside of the box, and send to:

Durham Geo Slope Indicator

Repair Department

Return Authorization: nnnnnnnn

12123 Harbour Reach Drive

Mukilteo, WA, USA 98275

## Limited Warranty

Durham Geo Slope Indicator warrants all products manufactured by it to be free of defects of workmanship and material for a period of one year from the date of delivery to the end-user. The obligation of Durham Geo Slope Indicator is hereafter limited to replacement or, at its option, repair of products returned to it with transportation charges to and from the Company paid by the customer (including prepayment of transportation charges to the Company) and which the company's examination shall disclose, to its satisfaction, were not free from such defects.

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# Operator's Manual

## SMARTROLL™ MP Handheld Instrument

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The presence of the Waste Electrical and Electronic Equipment (WEEE) marking on the product indicates that the device is not to be disposed via the municipal waste collection system of any member state of the European Union.

For products under the requirement of WEEE directive, please contact your distributor or local In-Situ Inc. office for the proper decontamination information and take back program, which will facilitate the proper collection, treatment, recovery, recycling, and safe disposal of the device.

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## Table of Contents

---

<b>1</b>	<b>Introduction</b>	<b>6</b>
	Scope	6
	Serial Number Location	6
<b>2</b>	<b>Safety</b>	<b>6</b>
<b>3</b>	<b>General Specifications</b>	<b>7</b>
<b>4</b>	<b>Sensor Specifications</b>	<b>8</b>
	Level, Depth, Pressure Sensor Specifications	8
	Barometric Pressure Sensor Specifications (Battery Pack)	8
	Conductivity Sensor Specifications	9
	Dissolved Oxygen RDO Fast Cap (Optical Sensor) Specifications	9
	ORP Sensor Specifications	10
	pH Sensor Specifications	10
	Air Temperature Sensor Specifications (Battery Pack)	10
	Sample Temperature Sensor Specifications (Probe)	11
<b>5</b>	<b>Battery Pack Specifications</b>	<b>12</b>
<b>6</b>	<b>Instrument Overview</b>	<b>13</b>
	Instrument Description	13
<b>7</b>	<b>System Components</b>	<b>13</b>
	Probe Dimensions with Restrictor On	14
	Probe Dimensions with Restrictor Off	14
	Sensors	15
<b>8</b>	<b>Probe Setup</b>	<b>16</b>
	Install the Batteries	16
	Installing the Sensors	18
<b>9</b>	<b>iSitu Overview</b>	<b>20</b>
	About the iSitu App	20
	Connect the Instrument to the iSitu App	20
	Live Readings Screen	23
	Change Parameters and Units	24
<b>10</b>	<b>iSitu Sites</b>	<b>25</b>
	About Sites	25
	Create a New Site	25
	Select a Site	27
	Edit a Site	27
	Delete a Site	28
	Restore a Site	28

---

---

<b>11 iSitu Data</b>	<b>29</b>
About Data	29
Record Data	29
View an Individual Reading	29
View and Email Data from the Selected Site	30
View, Email, or Delete Data from Any Site	32
Emailing Data From Different Screens in iSitu	34
Emailing from the Data screen	34
Emailing from the Logs screen	34
Emailing from the Readings screen	35
Emailing from the Data Details screen	35
Transfer Data to a Computer	35
Delete all Logs by Site	35
Restore Data	36
<b>12 iSitu Sensor Calibration</b>	<b>37</b>
About Calibration	37
Calibrate Multiple Sensors with Quick-Cal Solution	37
Calibrate the Rugged Dissolved Oxygen Sensor	39
Calibrate the Conductivity Sensor	42
Calibrate the Depth Sensor	44
Zero in Air	44
Setting the Depth Reference	45
Calibrate the pH Sensor	47
Calibrate the ORP Sensor	50
<b>13 Low-Flow Pump Testing</b>	<b>52</b>
Low-Flow Sampling with SMARTROLL™ MP Instrument	52
Purchase the Low-Flow App	52
Create a Low-Flow Template from the Desktop	53
Email Low-Flow Template to an iPod or iPhone	57
Load the Template into the iSitu App	57
Set up a Low Flow Test From a Template	59
Set up a Low-Flow Test without a Template	60
Install the Pump	64
Prepare the Flow Cell	64
Start a Low-Flow Test	65
Transfer Low-Flow Report to a Computer	65
<b>14 Care and Maintenance</b>	<b>67</b>
Maintenance Schedule	67

---

---

---

User-Serviceable Parts .....	67
O-rings .....	67
RDO Fast Sensor Cap Replacement .....	67
pH/ORP Sensor Replacement .....	67
Instrument Storage .....	67
Cleaning the pH/ORP Sensor .....	68
Remove Crystalline Deposits .....	68
Remove Oily or Greasy Residue .....	68
Remove Protein-Like Material or Slimy Film .....	69
Cleaning the RDO Sensor .....	69
Clean the Sensor Cap .....	69
Clean the Optical Window .....	69
Cleaning the Conductivity Sensor .....	69
Cleaning Procedure 1 .....	70
Cleaning Procedure 2 .....	70
Cleaning Procedure 3 .....	70
Cleaning Procedure 4 .....	70
<b>15 Declaration of Conformity .....</b>	<b>71</b>

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## Introduction

This manual is intended to describe the characteristics, operation, calibration, and maintenance of the SmarTROLL™ MP Instrument.

### Scope

This manual covers the following information.

Chapter 1—Introduction

Chapter 2—Safety

Chapter 3—Overview

Chapter 4—System Components

Chapter 5—Probe Setup

Chapter 6—iSitu Overview

Chapter 7—iSitu Sites

Chapter 8—iSitu Data

Chapter 9—iSitu Sensor Calibration

Chapter 10—Low-Flow Pump Testing

Chapter 11—Care and Maintenance

### Serial Number Location

The probe serial number is on the product label affixed to the probe body.

The battery pack serial number is on a sticker affixed to the battery pack casing.

## Safety

- Do not submerge the battery pack or the mobile display device in liquid.
- Ensure that the pH/ORP sensor is completely inserted into the port, so that no liquid can enter the instrument. The storage plug is not intended to be used when the instrument is deployed in water.
- Ensure that the RDO Sensor Cap is pressed firmly over the sensor lens and is flush with the instrument before submerging in liquid.
- Replace the cable if insulation or connectors are damaged.
- Make sure the probe and sensor O-rings are clean and free of damage.

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## General Specifications

Operating temperature	-5 to 50° C (23 to 122° F)
Storage temperature	-40 to 65° C (-40 to 149° F)
Dimensions	4.7 cm (1.85 in.) OD x 26.9 cm (10.6 in.) with restrictor installed (does not include connector)
Weight	694 g (1.53 lbs)
Wetted materials	PVC, 316 stainless steel, titanium, Acetal, Viton®, PC/PMMA
Environmental rating	IP68 with all sensors and cable attached. IP67 with sensors removed and cable detached.
Reading rate	1 reading every 10 seconds; data logged to smartphone.
Power	6 VDC from battery pack
Interface	iPhone® 4S, iPod touch® 5, or iPad® 3, 4, mini or later; iOS 6.0 or later. Bluetooth® Low Energy (BLE) radio. Purchase the iSitu™ App at the Apple® App Store.
Cable	Black polyurethane. Standard lengths available: 1.5 m, 4.6 m, 9.1 m, 30.5 m (5 ft, 15 ft, 30 ft, 100 ft)
Warranty	2-years
Notes	Specifications are subject to change without notice. Apple, iPhone, iPod touch, and iPad are trademarks of Apple Inc. registered in U.S. and other countries. Bluetooth is a registered trademark of Bluetooth SIG, Inc. Viton is a registered trademark of DuPont Performance Elastomers L.L.C.

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## Sensor Specifications

### Level, Depth, Pressure Sensor Specifications

Accuracy	Typical $\pm 0.1\%$ FS @ 15° C; $\pm 0.3\%$ FS max. from 0 to 50° C
Range	76 m (250 ft); absolute (non-vented)
Resolution	$\pm 0.01\%$ FS or better
Sensor Type	Fixed
Response Time	Instantaneous in thermal equilibrium
Units of Measure	Pressure: psi, kPa, bar, mbar, mmHg, inHg Level: mm, cm, m, in, ft
Methodology	Piezoresistive; ceramic

### Barometric Pressure Sensor Specifications (Battery Pack)

Accuracy	$\pm 3$ mbar max.
Range	300 to 1100 mbar
Resolution	0.01 mbar
Sensor Type	Fixed
Response Time	Instantaneous in thermal equilibrium
Units of Measure	psi, kPa, bar, mbar, mmHg, inHg, Torr, atm
Methodology	Piezoresistive pressure sensor



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## Conductivity Sensor Specifications

Accuracy	Typical $\pm 0.5\%$ + 1 $\mu\text{S}/\text{cm}$ ; $\pm 1\%$ max.
Range	5 to 100,000 $\mu\text{S}/\text{cm}$
Resolution	0.1 $\mu\text{S}/\text{cm}$
Sensor Type	Fixed
Response Time	Instantaneous in thermal equilibrium
Units of Measure	Actual conductivity ( $\mu\text{S}/\text{cm}$ , $\text{mS}/\text{cm}$ ) Specific conductivity ( $\mu\text{S}/\text{cm}$ , $\text{mS}/\text{cm}$ ) Salinity (PSU) Total dissolved solids (ppt, ppm) Resistivity (Ohms-cm) Density ( $\text{g}/\text{cm}^3$ )
Methodology	Std. Methods 2510 EPA 120.1

## Dissolved Oxygen RDO Fast Cap (Optical Sensor) Specifications

Accuracy	$\pm 0.1$ mg/L; $\pm 0.2$ mg/L; $\pm 10\%$ of reading
Range	0 to 8 mg/L; 8 to 20 mg/L; 20 to 50 mg/L; Full operating range: 0 to 50 mg/L
Resolution	0.01 mg/L
Sensor Type	Fixed with replaceable RDO Fast Cap (life: 1 year typical)
Response Time	T90: <30 sec. T95: <45 sec.
Units of Measure	mg/L, % saturation, ppm
Methodology	EPA-approved In-Situ Methods 1002-8-2009 1003-8-2009 1004-8-2009

---

## ORP Sensor Specifications

Accuracy	±5.0 mV
Range	±1400 mV
Resolution	0.1 mV
Sensor Type	Replaceable pH/ORP combo sensor
Response Time	<15 sec.
Units of Measure	mV
Methodology	Std. Methods 2580

## pH Sensor Specifications

Accuracy	±0.1 pH unit from 0 to 12 pH units
Range	0 to 14 pH units
Resolution	0.01 pH unit
Sensor Type	Replaceable pH/ORP combo sensor
Response Time	<15 sec., pH 7 to pH 4
Units of Measure	pH units
Methodology	Std. Methods 4500-H+ EPA 150.2

## Air Temperature Sensor Specifications (Battery Pack)

Accuracy	±2° C
Range	-20 to 70° C (-4 to 158° F)
Resolution	0.1° C
Sensor Type	Fixed
Response Time	<30 sec.
Units of Measure	Celsius, Fahrenheit
Methodology	EPA 170.1

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## Sample Temperature Sensor Specifications (Probe)

Accuracy	±0.1° C
Range	-5 to 50° C (23 to 122° F)
Resolution	0.01° C or better
Sensor Type	Fixed
Response Time	<30 sec.
Units of Measure	Celsius, Fahrenheit
Methodology	EPA 170.1

---

## Battery Pack Specifications

Battery Type	Four 1.5V AA lithium or alkaline batteries
Operating temperature	-5 to 50° C (23 to 122° F); 95% relative humidity, non-condensing
Storage temperature	-40 to 65° C (-40 to 149° F); 95% relative humidity, non-condensing
Dimensions & weight	9.5 x 7.6 x 5.7 cm (3.75 x 3 x 2.25 in.) (H x D x W). Weight: 165 g (5.8 oz)
Materials	PC/ABS
Environmental rating	IP67 with battery cover closed
Output options	BLE radio
Battery type	4 AA Lithium or Alkaline
Warranty on battery pack	1-year
Warranty on cable	1-year

---

## ***Instrument Overview***

### **Instrument Description**

The smarTROLL™ MP Handheld Instrument is comprised of a mobile display, battery pack, cable, and multiparameter water quality probe. The optical Rugged Dissolved Oxygen (RDO®), conductivity, pressure, and temperature sensors are integrated into the probe. The pH/ORP and the RDO Sensor Cap are replaceable.

### ***System Components***

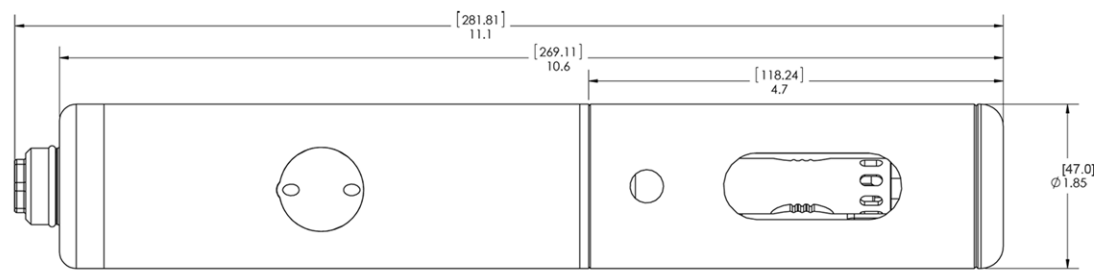
The system includes the following components.

- Integrated sensors: RDO, conductivity, pressure, and temperature
- Plug-in pH/ORP sensor
- RDO Fast Sensor Cap
- Stainless steel restrictor
- Calibration cup
- Battery pack and cable

#### **Accessories Purchased Separately**

- Replacement RDO Fast Sensor Cap
- Replacement pH/ORP sensor
- Calibration Kit (includes calibration cup, 3 sponge wafers, vented cap, and storage cap)
- Cable 1.5 m (5 ft), 4.6 m (15 ft), 9.1 m (30 ft) and 30.5 m (100 ft).
- Maintenance kit
- Replacement battery pack
- Storage/Calibration cup
- Low-Flow kit
- iPod® Touch (for instrument control and data display)
- iTunes® account for transferring data files as an alternate to email

Probe Dimensions with Restrictor On



Total length with connector	281.81 mm (11.1 in.)
Total length without connector	269.11 mm (10.6 in.)
Restrictor length	118.24 mm (4.7 in.)
Diameter	47 mm (1.85 in.)

Probe Dimensions with Restrictor Off

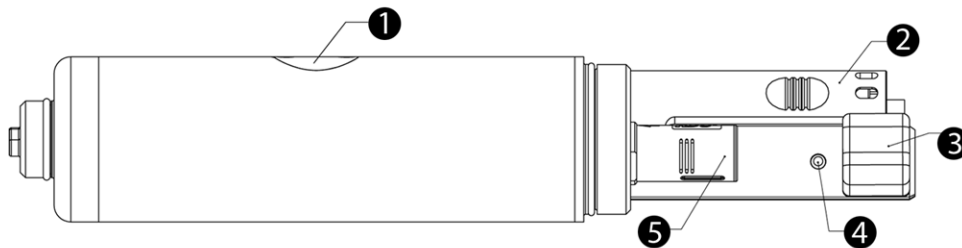


Sensor length	81.09 mm (3.2 in.)
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## Sensors

Sensors include optical RDO (Rugged Dissolved Oxygen), pH/ORP, conductivity, pressure, and temperature.



1	Pressure sensor 76 m (250 ft)
2	pH/ORP sensor
3	Conductivity sensor
4	Temperature sensor
5	RDO Sensor

---

## Probe Setup

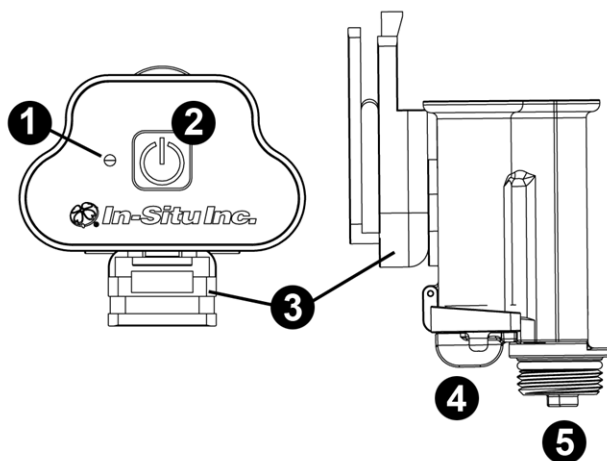
The probe is shipped with a storage plug and protective dust caps in place.



- |   |   |
|---|---|
| 1 | Dust cap protector on the RDO Sensor. (Install the RDO Cap before deploying the instrument.)                  |
| 2 | pH/ORP storage plug. (Remove the storage plug and install the pH/ORP sensor before deploying the instrument.) |

## Install the Batteries

The 4 AA batteries that are shipped with the battery pack are likely to last for 80 hours of continuous use.



- |   |                           |
|---|---------------------------|
| 1 | Power indicator           |
| 2 | On/Off button             |
| 3 | Belt clip                 |
| 4 | Battery compartment latch |
| 5 | Cable connection          |



- 
1. Twist the cable connector counterclockwise to remove the cable from the battery pack.
  2. Slide the lever on the battery compartment to release the cover.



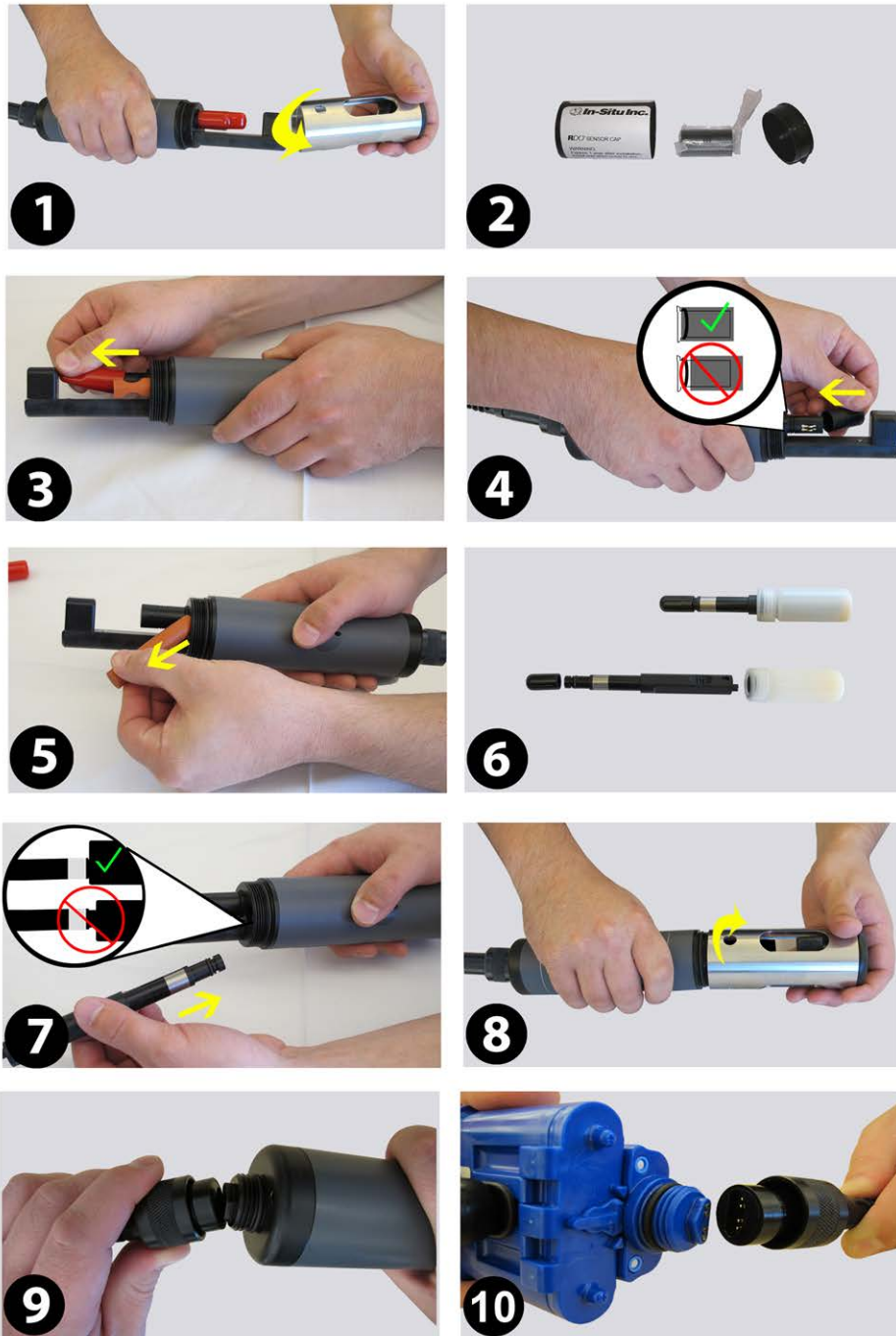
3. Install the 4 AA batteries according to the +/- indicators engraved on the outside cover.



4. Close the cover and slide the lever to lock the compartment.

---

## Installing the Sensors



1. Twist the restrictor off the probe.
2. Locate the RDO Sensor Cap container and remove the cap.
3. Remove the dust cap from the RDO Sensor.
4. Align the flat edge of the RDO Sensor with the slotted edge of the RDO Cap and press the cap into position. Push until the cap is firmly in place.



Important: Avoid touching the sensor lens and the sensing material on the top of the cap.

5. Remove the orange plug from the pH/ORP port.
6. Remove the pH/ORP sensor from the storage bottle. Keep the bottle for future sensor storage.
7. Use the alignment marks to properly align the pH/ORP sensor with the port connection, and press firmly into place. Push until the sensor is completely inserted into the port.
8. Twist the restrictor onto the probe.
9. Align the pins on the cable with the pins on the probe, then twist the outer portion of the connector until the connection is secure.
10. Align the pins on the cable with the pins on the battery pack, then twist the outer portion of the connector until the connection is secure.



Important: The RDO Sensor Cap and pH/ORP sensor must be installed firmly in place to prevent water from entering the instrument.

---

## *iSitu Overview*

### About the iSitu App

The iSitu App is the user interface and control application for In-Situ Inc. handheld water quality instruments. You can use iSitu on the Apple iPod Touch, and iPhone for up to five devices per purchased license.

iSitu allows you to accomplish the following tasks.

- View live readings that update every 10 seconds.
- Change parameters and units.
- Record data.
- Email data in spreadsheet format.
- Transfer data from mobile device to a computer.
- Organize data by Site location.
- Calibrate Sensors and View Reports
- Conduct Low-Flow Pump Testing (Additional purchase is required.)

### *Estimated iPod Battery Life*

The table below shows the estimated battery life for the iPod. The values are dependent on the number of readings taken and the brightness setting on the display. To change brightness settings, see **Settings > Brightness & Wallpaper** on the iPod.

BRIGHTNESS					NUMBER OF READINGS	BATTERY TIME (HOURS)
MIN	1/4	1/2**	3/4	FULL		
X					2,500	6.9
	X				1,950	5.4
		X			1,700	4.7
			X		1,500	4.2
				X	1,050	3.3

\*Values provided assume location services and WiFi enabled. Disabling these features can provide an additional 0.5 to 1 hour of life.

\*\*Default


### Connect the Instrument to the iSitu App

1. Make sure that the cable is connected to the instrument and the battery pack.
2. Press the power button on the battery pack.
3. On the mobile device, tap **Settings**.



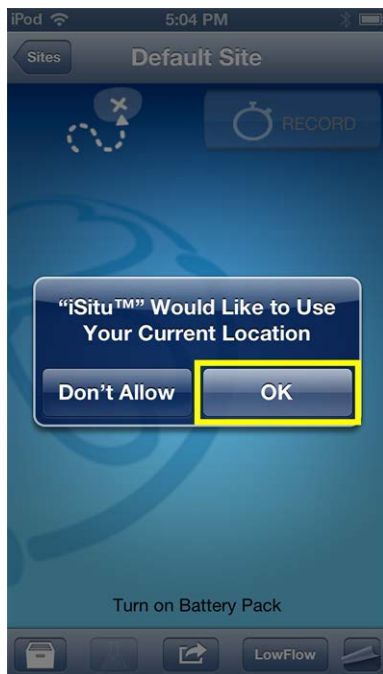
4. Turn Bluetooth on.



5. Press the Home button (round button on the mobile device frame) to show all apps.
6. Tap the iSitu icon  to open the iSitu App.
7. If you are prompted to allow iSitu to use your current locations, tap **OK**.



If you allow iSitu to use your current locations it will enable the mapping feature for site setup. If you select **Don't Allow**, you can change the setting later. **See Settings > Privacy Settings > Location Services.**



8. Live readings appear on screen.



---

## Live Readings Screen

The Live Readings screen is also referred to as the Home screen. If you touch the **Home** icon within the app you will return here.



Live readings update every 10 seconds.



1	Sites button - setup sites, select another site
2	Site photo (optional)
3	Record/Stop button - records sensor readings every 10 seconds
4	Sensor readings - updated every 10 seconds
5	Data files - access files stored on the mobile device
6	Sensor calibration
7	View or email data
8	Access Low-Flow (additional purchase required)
9	Help

---

## Change Parameters and Units

1. From the **Home** screen, tap any parameter field.



2. Swipe the left side of the parameter pick wheel to find the appropriate parameter.
3. Swipe the right side of the parameter pick wheel to select the appropriate unit.
4. Tap the **Set** button to set the parameter and unit selection.



---

## iSitu Sites

### About Sites

A site represents the physical location at which the instrument collects data. For example, you can create a site to represent a lake, gauging station, well, tank, number, or nearby landmark.

If you do not set up a site, your data will be associated with **Default Site**. The site name is displayed at the top of the **Live Readings** screen.

Tap the **Sites** button to select or edit an existing site, or to create a new site.

### Create a New Site

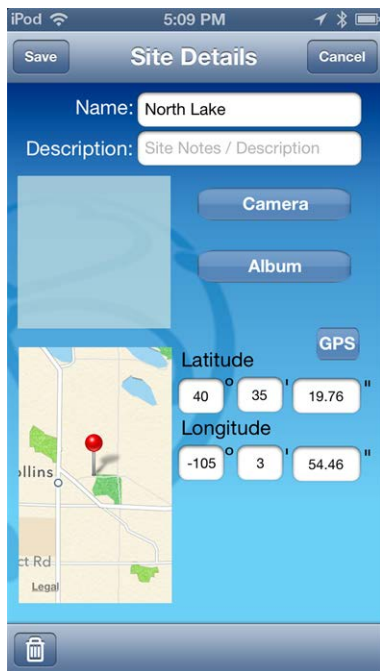
1. From the **Live Readings** screen, tap the **Sites** button.



2. A list of existing sites appears.



3. Tap the **New Site** button. The **Site Details** screen appears.



4. Tap the **Name** field. Type the name for the new site and tap **Return**.
5. To add a description, tap the **Description** field. Type a description and tap **Return**. A description is optional.
6. To take a site photo, tap the **Camera** button, tap the camera icon to take a new photo, tap the **Use** button. A site photo is optional.
7. To select an existing photo, tap the **Album** button, tap **Camera Roll**, tap an existing photo.

8. To locate your site with Maps or GPS, tap the **GPS** button and your current location is automatically associated with the site. You can also enter GPS coordinates, or tap and hold on the map to select a location.



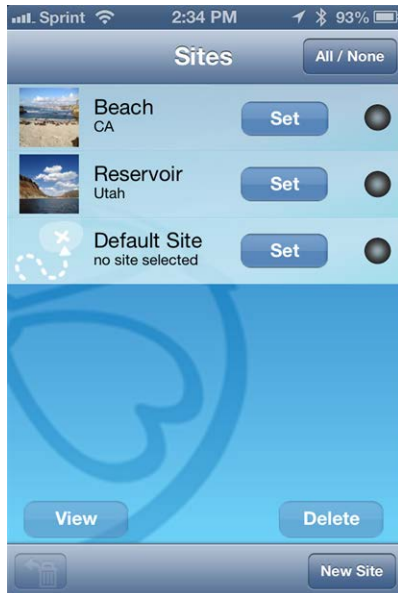
Location Services must be turned on for an accurate location to display on the map. See **Settings > Location and Security**.

9. Tap the **Save** button.
10. Tap the **Set** button next to the site you created. Now you are ready to record data associated with the selected site.

## Select a Site

After a site has been created, you can select it to record data that will be associated with that site.

1. From the Live Readings screen, tap the **Sites** button.
2. Locate the site with which you want to associate your data.



3. Tap the **Set** button. The site will appear on the Live Readings screen and recorded data will be associated with the selected site.

## Edit a Site

1. Tap the **Sites** button.
2. Locate the site you intend to edit.
3. Tap the **circle** next to the **Select** button for the site.
4. Tap the **View** button, and make changes to the site information.
5. Tap the **Save** button.

---

## Delete a Site

1. Tap the **Sites** button.
2. Locate the site you intend to delete.
3. Tap the **circle** associated with the site.
4. Tap the **Delete** button.



This procedure sends the site to the trash where you can choose to completely delete it, or restore the site. You cannot delete the default site.

## Restore a Site

1. It is possible to restore a deleted site.
2. From the **Home** screen, tap the **Sites** button.
3. Tap the **Restore From Garbage Can** icon.



4. Tap the site you intend to restore.
5. Tap the **Restore** button.

---

## *iSitu Data*

### About Data

iSitu allows you to view real-time readings, record readings in ten-second intervals, email data, store data to the mobile device, and transfer data from the mobile device to a PC.

### Record Data

1. Tap the **Record** button on the **Live Readings** screen to record data. The number on the stopwatch icon represents how many 10-second data intervals have transpired.



2. To stop recording, tap **Stop**.
3. Now you can email the data or download it to a computer.

### View an Individual Reading

Recorded data is stored on the Apple device in a comma-separated value (CSV) file and can be viewed in a spreadsheet format after the file has been emailed from the mobile device, or transferred to a computer via iTunes.

1. To view an individual reading, tap the **Action** icon.



2. Tap **View Last Reading**.



3. The most recent data in the last ten-second interval appears. Tap the **Home** icon to return to the **Live Readings** screen or tap the **Envelope** icon to email the data.

## View and Email Data from the Selected Site

After you have recorded data, you can email the data as a CSV file that can be opened with common spreadsheet software. Make sure the email feature is enabled on the mobile device.



See **Settings > Mail, Contacts, Calendars > Add Account**. You must also have connection to WiFi or cell phone service if you are using an iPhone®. See **Settings > Wi-Fi**.

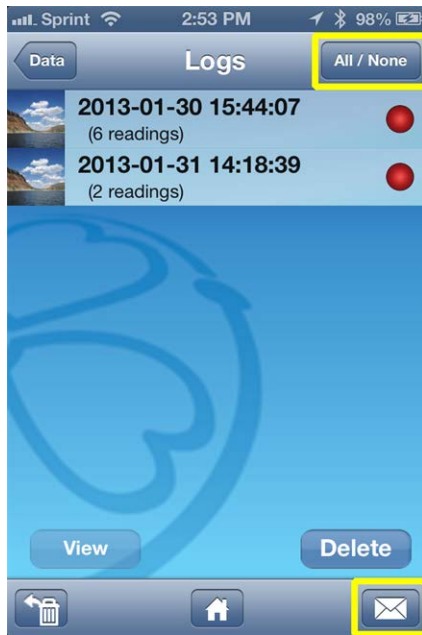
1. Tap the **Action** icon.



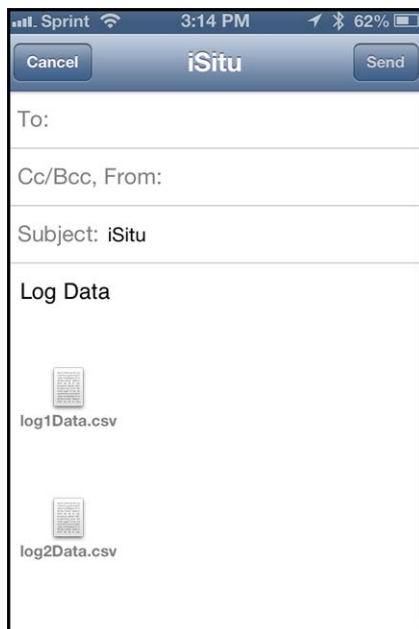
2. Tap **View Log List**. This shows a list for only the selected site.



3. To select all logs in the list, tap the **All/None** button, or to select individual logs, tap them separately.



4. Tap the **Envelope** icon.
5. An email form appears with the logs that were selected attached.



6. Enter an email address in the **To:** field.
7. Tap the **Send** button.



You can also transfer data to a computer using iTunes.

## View, Email, or Delete Data from Any Site

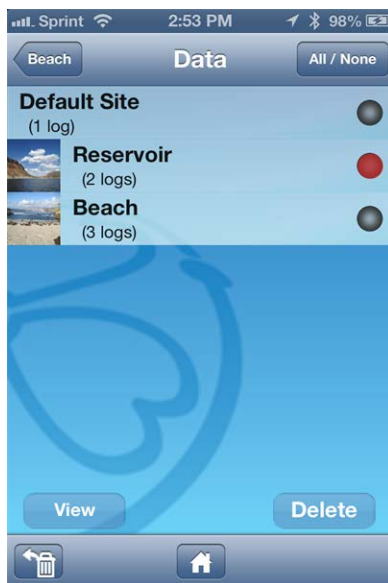
After you have recorded data, you can email the data as a CSV file that can be opened with common spreadsheet software. Make sure the email feature is enabled on the mobile device. See **Settings > Mail, Contacts, Calendars > Add Account**. You must also have connection to WiFi. See **Settings > Wi-Fi**.



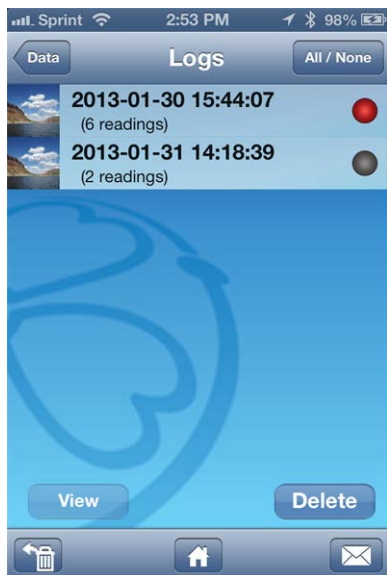
1. Tap the **Data** icon.



2. The Data screen displays a list of sites and the number of data logs within each site. Tap the site that contains the data you want to view, email or delete. The selection circle turns red and the **View**, and **Delete**, buttons become active.



3. Tap the **View** button.
4. The list of logs associated with that site appears. Tap the log you want to view. The selection circle turns red and the **View** and **Delete** buttons become active. The **Envelope** icon also becomes active. You can select any of these options.



5. Tap the **View** button.
6. The list of readings within the log appears. Tap the reading you want to view. The selection circle turns red and the **View** button becomes active.
7. Tap the **View** button. The data for an individual reading appears.



## Fort Collins

at 2013-06-03 12:27:18

<i>Parameter</i>	<i>Value</i>	<i>Unit</i>	<i>Quality</i>
Baro	839.5	mbar	OK: Normal
Temp	25.28	C	OK: Normal
RDO	6.48	mg/L	OK: Normal
RDO Sat	95.7	%	OK: Normal
Air Temp	27.40	C	OK: Normal



## Emailing Data From Different Screens in iSitu

### Emailing from the Data screen

Select one or more sites and email all logs associated with the selected sites.

### Emailing from the Logs screen

Select one or more logs (from a single site) and email the selected logs.

---

## Emailing from the Readings screen

Select one or more readings (from a single log) and email them as one file. The file name will be appended with the word "reading."

## Emailing from the Data Details screen

Emailing from the Data Details view will email all readings in that log

## Transfer Data to a Computer

1. Connect the mobile device to a computer with iTunes installed.
2. Click on the Apple device icon next to the eject button.



3. Click the word **Apps** near the top of the screen.
4. Scroll to the bottom of the screen and click on **iSitu**.
5. Click on a file and drag it to your desktop.



You can also email data to your computer.

## Delete all Logs by Site

1. Tap the **Data** icon.



2. Tap the **All/None** button, or tap an individual site. The selection circle turns red when a site is selected.
3. Tap the **Delete** button. All logs associated with the site will be deleted.



This procedure sends the logs to the trash where you can choose to completely delete them or restore the logs.

## Restore Data

It is possible to restore deleted logs.

1. Tap the **Data** icon.
2. Tap the **Restore from Garbage Can** icon.



3. The contents of the Trash Can are displayed.
4. Tap the **All/None** button, or tap the individual logs you want to restore.
5. Tap the **Restore** button.



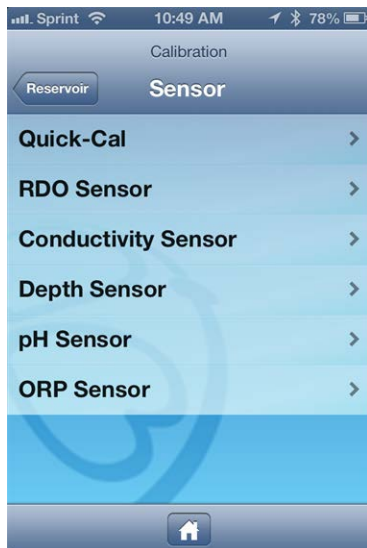
If you want to permanently delete data from the Trash Can, tap the **Delete** button.

---


## *iSitu Sensor Calibration*

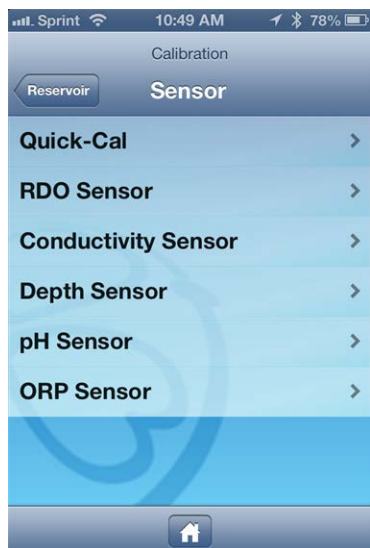
### About Calibration

Tap the **Calibration** icon  in the iSitu App to access a list of sensors that are available for calibration.



### Calibrate Multiple Sensors with Quick-Cal Solution

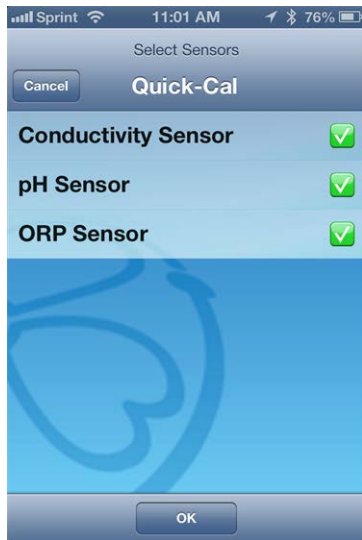
1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **Quick-Cal**.



3. The conductivity, pH, and ORP sensors are automatically selected. Tap the green check mark next to a sensor if you want to exclude it from the quick calibration.



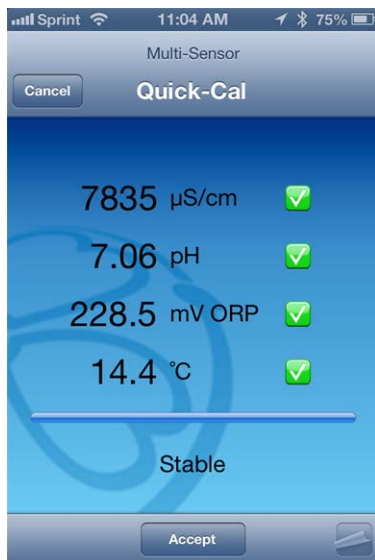
The dissolved oxygen sensor cannot be calibrated using the Quick-Cal procedure.



4. Tap **OK**.
5. Fill the calibration cup to the fill line with Quick-Cal solution.
6. Place the instrument into the calibration cup, and tap **Start**.



7. When the calibration is stable, tap the **Accept** button.

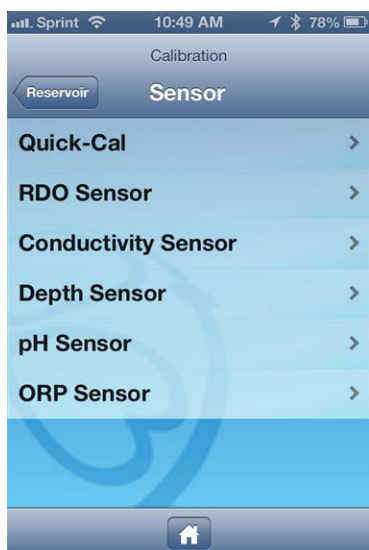


8. Rinse the sensors with DI water.

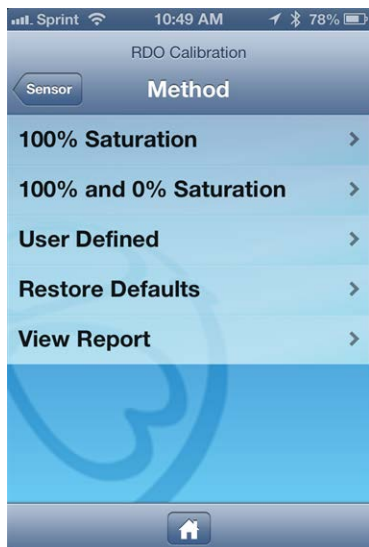
## Calibrate the Rugged Dissolved Oxygen Sensor

The RDO sensor requires very little maintenance. The zero oxygen calibration is optional.

1. Tap the **Calibration** icon .
2. Tap **RDO Sensor**.



3. Select the method by which you intend to calibrate the sensor. This example demonstrates a two-point calibration. Tap **100% and 0% Saturation**.



4. Place a water-saturated sponge in the bottom of the calibration cup. Place the instrument into the calibration cup, and tap **Start**.

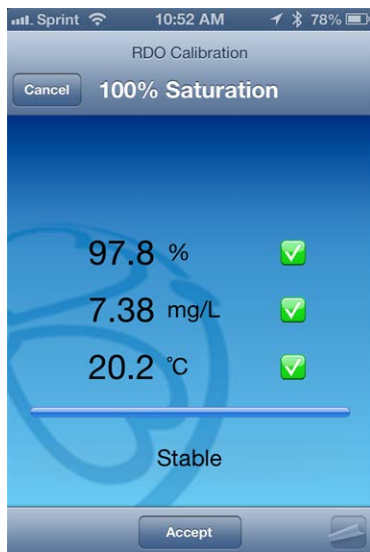


The calibration cup must be vented to barometric pressure. If you are using the calibration cup pictured below, make sure the vented cap is installed. If you are using the twist-on storage cup, set the instrument in the cup, but do not twist it into place.



5. When the calibration is stable, tap the **Accept** button.





6. Remove the sponge and add fresh sodium sulfite solution to the fill line. Place the instrument into the calibration cup, and tap **Start**.




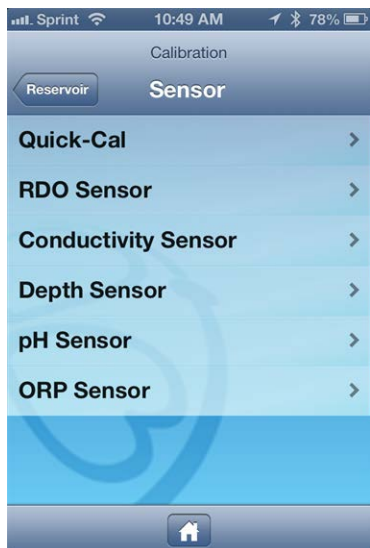
7. When the calibration is stable, tap the **Accept** button.



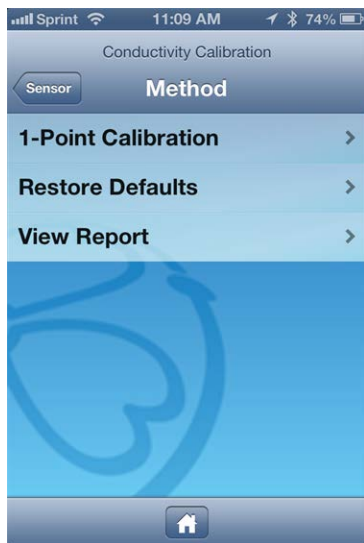
8. To view the calibration report, tap **View Report**.
9. Rinse the sensors thoroughly with DI water.

## Calibrate the Conductivity Sensor

1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **Conductivity Sensor**.



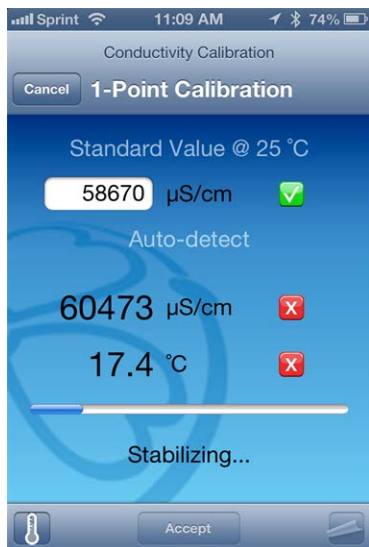
3. Tap 1-Point Calibration



4. Make sure the vented cap is installed on the calibration cup. Fill the cup to the fill line with calibration standard. Place the instrument into the calibration cup, and tap **Start**.



5. iSitu automatically detects the calibration standard.




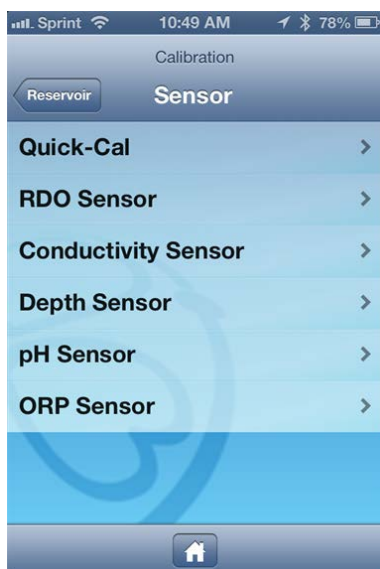
If your calibration standard references 20° C, tap the **Thermometer** icon and change the reference temperature.

6. Once the calibration is stable, tap the **Accept** button.
7. To view the calibration report, tap **View Report**.
8. Rinse the sensors with DI water.

## Calibrate the Depth Sensor

### Zero in Air

1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **Depth Sensor**.



3. Tap **Zero in Air**.




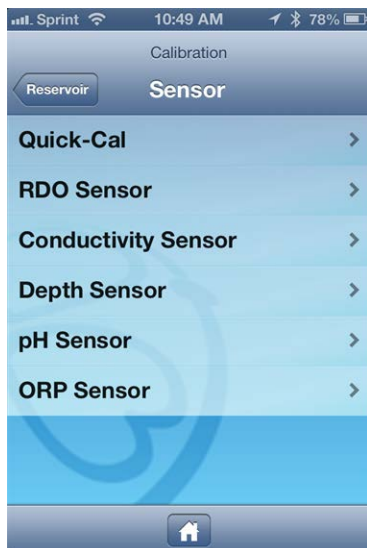
Do not perform a "Zero in Air" calibration if a Depth reference is already set because it will result in a faulty calibration. To clear a Depth reference, tap **Restore Defaults**.



4. Ensure that the pressure sensor is exposed to air and is not submerged in liquid.
5. Tap the **Start** button.
6. When the calibration is stable, tap the **Accept** button.

### Setting the Depth Reference

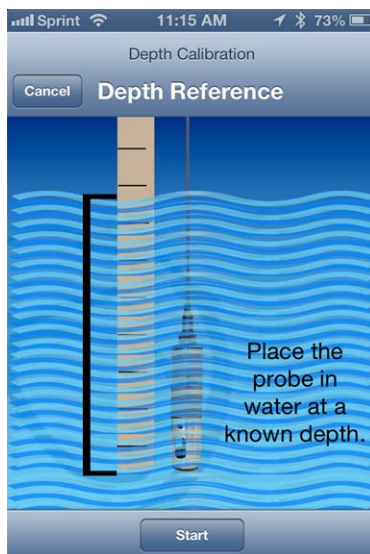
1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **Depth Sensor**.



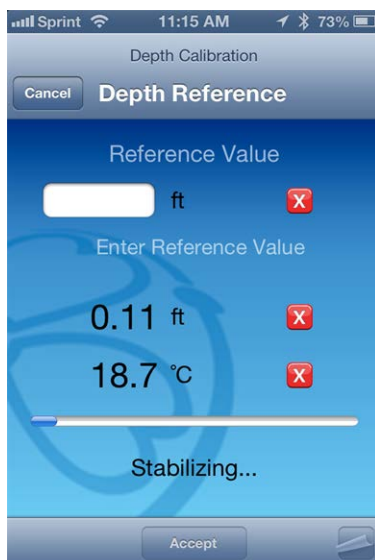
3. Tap **Depth Reference**.



4. Place the instrument in the water at a known depth, and tap the **Start** button.



5. Tap the **Reference Value** field and enter the value of the known depth reference.






A depth reference applies an offset equal to the distance from the pressure sensor to a desired location, such as the bottom of the probe, so that the depth reading is reported from the desired location, rather than from the location of the pressure sensor.

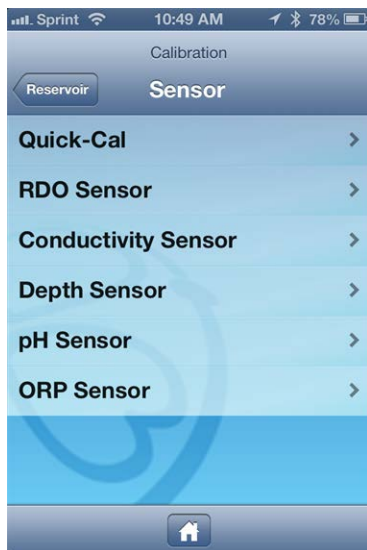


1	Pressure sensor
2	Example of a Depth reference setting

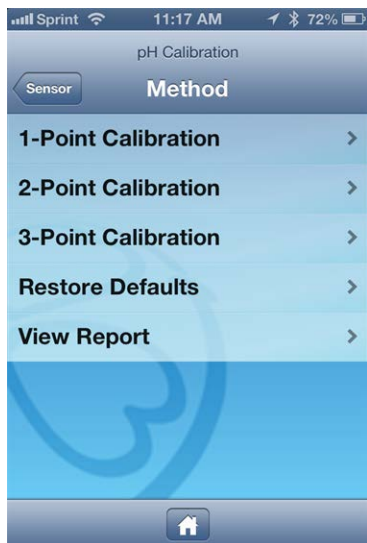
6. When the calibration is stable, tap the **Accept** button.

## Calibrate the pH Sensor

1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **pH Sensor**.



3. Select the method by which to calibrate the sensor. This example demonstrates a two-point calibration. Tap **2-Point Calibration**.

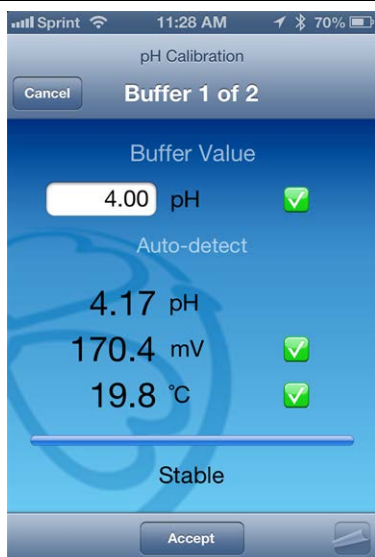


4. Make sure the vented cap is installed on the calibration cup. Fill the cup to the fill line with the first calibration buffer. Place the instrument into the calibration cup, and tap **Start**.

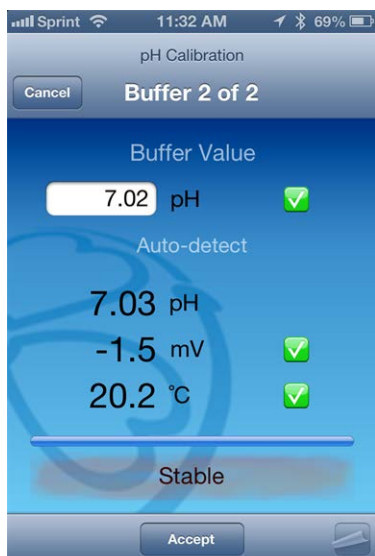


5. When the calibration is stable, tap the **Accept** button.






6. Fill the cup to the fill line with the second calibration buffer. Place the instrument into the calibration cup, and tap **Start**.
7. When the calibration is stable, tap the **Accept** button.

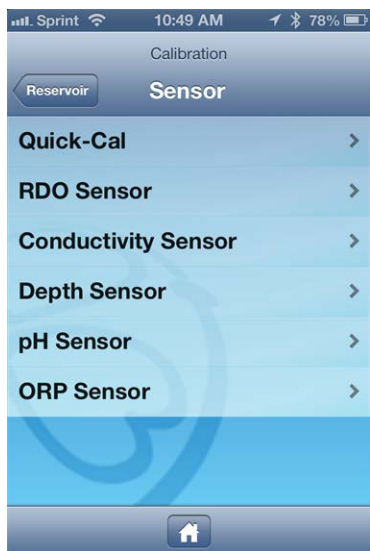


8. To view the calibration report, tap **View Report**.

---

## Calibrate the ORP Sensor

1. Tap the **Calibration** icon  to access a list of sensors that are available for calibration.
2. Tap **ORP Sensor**.



3. Tap 1-Point Calibration



4. Make sure the vented cap is installed on the calibration cup. Fill the cup to the fill line with calibration standard. Place the instrument into the calibration cup, and tap **Start**.



5. When the calibration is stable, tap the **Accept** button.
6. To view the calibration report, tap **View Report**.

---

## Low-Flow Pump Testing

### Low-Flow Sampling with SMARTROLL™ MP Instrument


Low-Flow sampling allows you to automate the collection of well and pumping information, monitor and record the stabilization of key water quality parameters, and automatically generate sample reports that conform to federal and regional regulations.

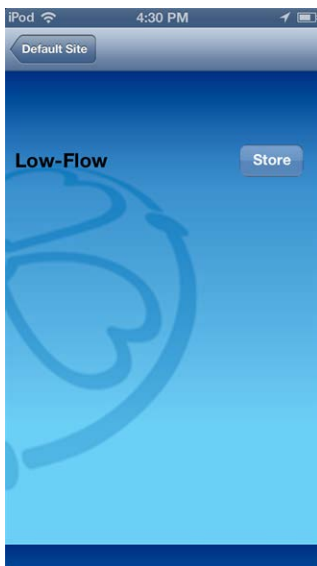
You can set up a template in the office and email it to a technician, or the setup can be done entirely in the field.

You need the following equipment.

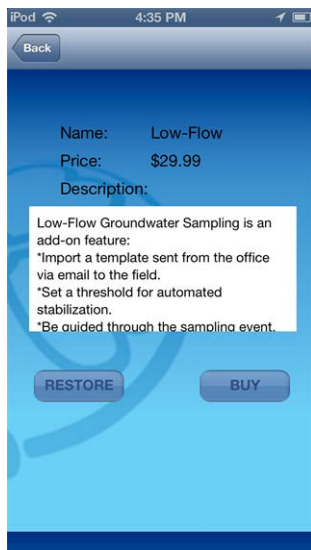
- Pump and tubing
- Flow cell, fittings, and base plate or stake
- SMARTROLL MP Instrument, battery pack, and cable
- iPod® or iPhone® loaded with the iSitu App
- Optional turbidity meter

### Purchase the Low-Flow App

1. Tap the **Gear** icon .
2. Tap the **Store** button.



3. Tap the **Buy** button. You are asked to confirm your purchase. Tap the **Buy** button again.



4. You are prompted to enter your iTunes information. The Low-Flow App will be accessible the next time you tap the **Gear** icon.

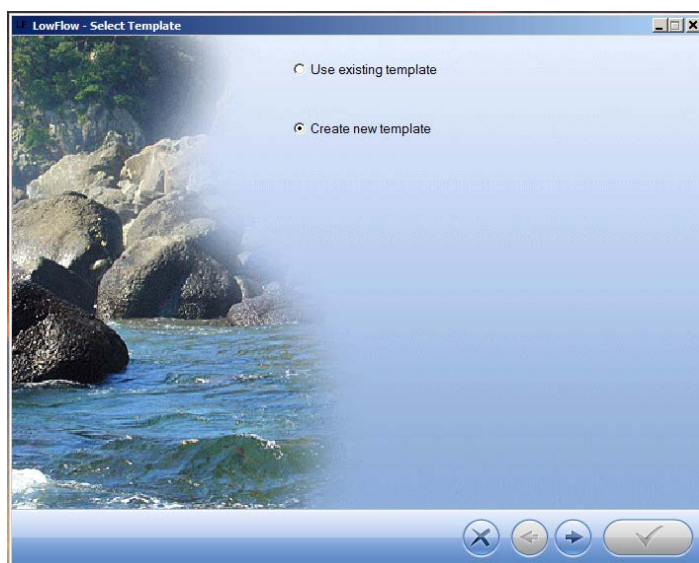
## Create a Low-Flow Template from the Desktop

You can create a Low-Flow template from a desktop or laptop computer and email it to technicians in the field.

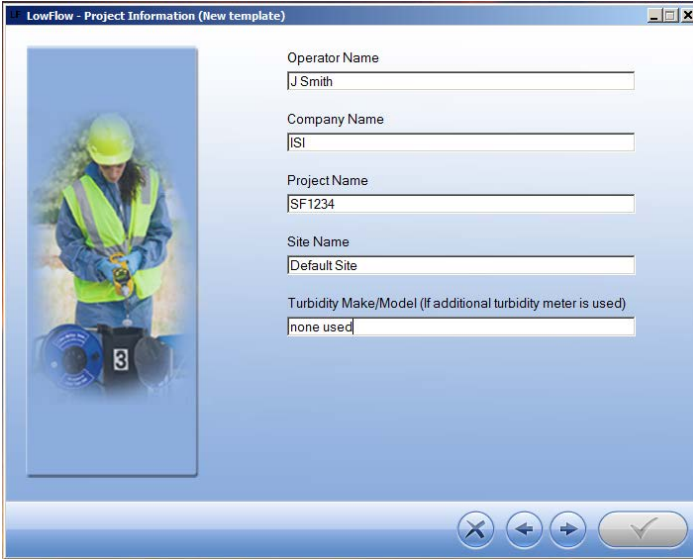


This Windows® software application is designed to work in conjunction with the iSitu mobile App and the SMARTROLL MP Instrument.

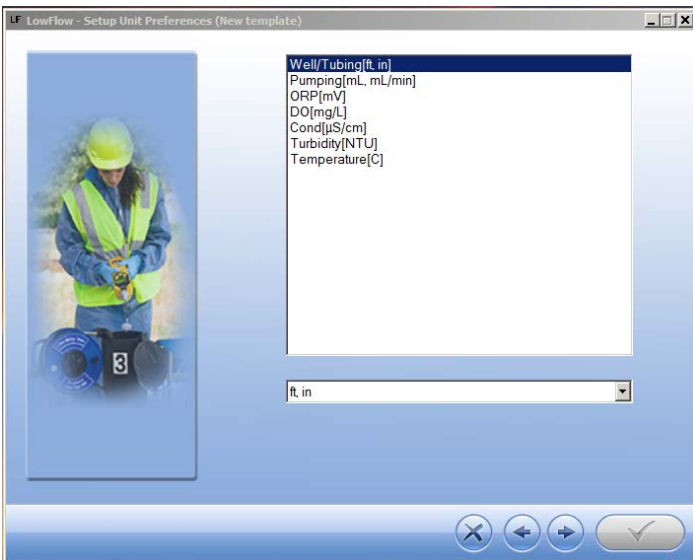
1. Open the Low-Flow software from within Win-Situ 5. When you are asked if you would like to connect to the device, click **No**.
2. Click the **Tools** menu and select **LowFlow Setup**.



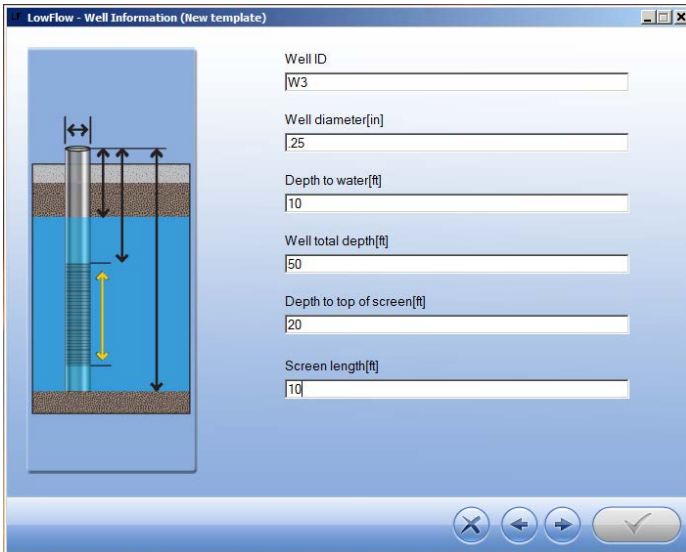
3. Click **Create New Template**, and click the **right arrow** button. The **Project** screen appears.



4. Enter project information and click the **right arrow** button. The **Units** screen appears.



5. If necessary, select a parameter and then click the drop-down box to select units.
6. Click the **right arrow** button. The **Well Information** screen appears.



LowFlow - Well Information (New template)

Well ID  
W3

Well diameter[in]  
25

Depth to water[ft]  
10

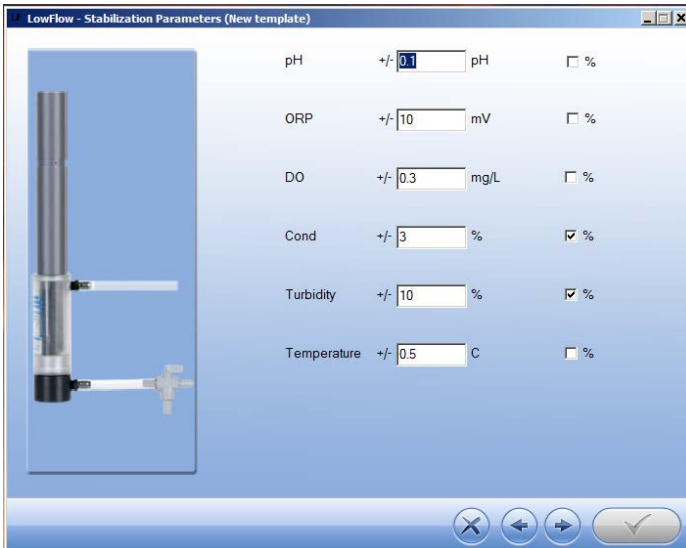
Well total depth[ft]  
50

Depth to top of screen[ft]  
20

Screen length[ft]  
10

Navigation buttons: [X] [Left Arrow] [Right Arrow] [Checkmark]

7. When you click on a field, the corresponding area of the well diagram is highlighted. Enter the well information.
8. Click the **right arrow** button. The **Stabilization Parameters** screen appears.



LowFlow - Stabilization Parameters (New template)

pH +/- 0.1 pH ☐ %

ORP +/- 10 mV ☐ %

DO +/- 0.3 mg/L ☐ %

Cond +/- 3 % ☒ %

Turbidity +/- 10 % ☒ %

Temperature +/- 0.5 C ☐ %

Navigation buttons: [X] [Left Arrow] [Right Arrow] [Checkmark]

9. The default stabilization criteria is shown. A selected percentage checkbox indicates that the stabilization criterion for the parameter is based on percentage rather than an absolute value.
10. Edit the information if necessary, and click the **right arrow** button.

LowFlow - Pump Information (New template)

Pump model/type  
P200

Internal pump volume[gal]  
5

Tubing type  
Silicone

Tubing inner diameter[in]  
.25

Tubing length[ft]  
6

Pump placement from top of well casing[ft]  
30

Total system volume[gal]  
0.54

11. Enter the pump information. The total system volume is calculated using the internal pump volume, the tubing inner diameter, and the tubing length.
12. Click the **right arrow** button. The **Purging Information** screen appears.

LowFlow - Purging Information (New template)

Pumping rate[mL/min]  
1

System purge time[min:sec] (Range: 00:10 to 99:59)  
90:00

Sample rate[min:sec] (Range: 00:10 to 99:59)  
90:00

☒ Auto calculate sample rate using system purge time

Drawdown estimate[in]

13. Enter the pumping rate you intend to use during the test.
14. Enter the sampling rate you intend to use, or select **Auto calculate sample rate using system purge time** if you want the software to assign a sample rate.
15. Enter an estimate of the drawdown you expect during the testing.
16. Click the **check mark**. You can save the template with the default name or enter a different name.



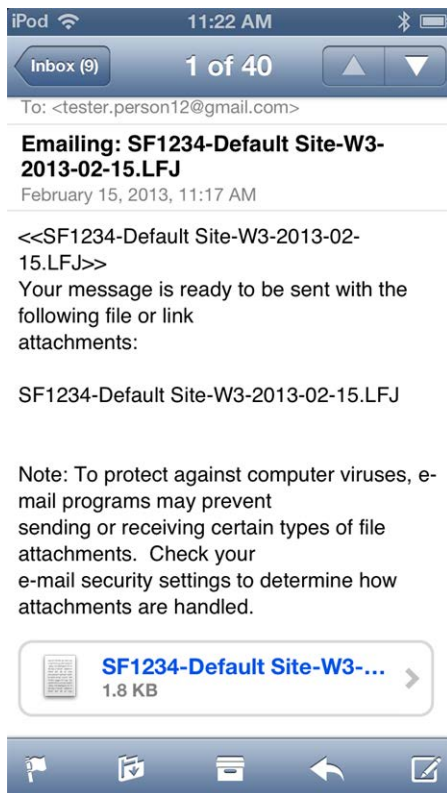
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## Email Low-Flow Template to an iPod or iPhone

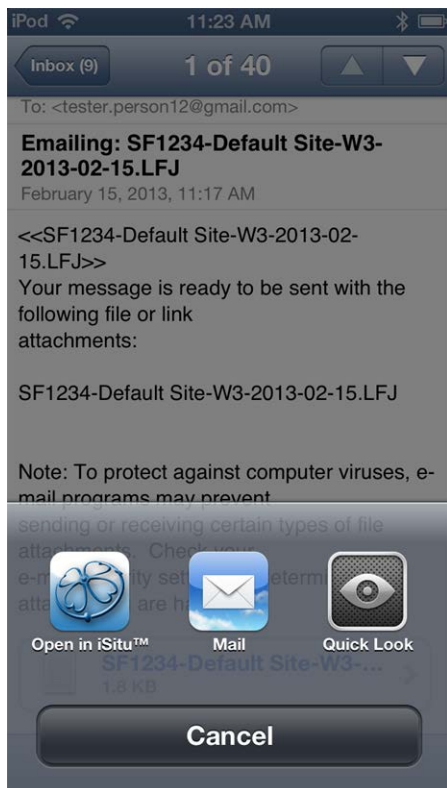
1. Low-Flow templates are by default saved to your computer in **My Documents/LowFlow Templates**.
2. One way to email a template is to open the **LowFlow Templates** folder and right-click on the template you want to email.
3. Select **Send to** and select **Mail recipient**. A new email will open with the template attached.
4. Send the email to a device that has email enabled.

## Load the Template into the iSitu App

1. Open the email on the mobile device.
2. Scroll to the attached template.



3. Tap and hold the attachment. A pop-up menu appears.




4. Tap the **iSitu icon**. The iSitu app opens with the template loaded. If you would like to save the template to the device, tap the **Save** button.



You can swipe through the screens and edit information before you save changes to the template.

## Set up a Low Flow Test From a Template

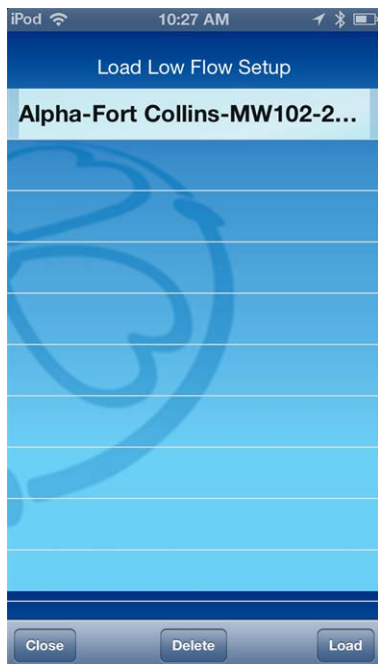
1. Tap the **Gear** icon  to access the Low-Flow section of the App.



2. The **Project** screen appears.




3. Tap the **Template** button. A list of all saved templates appears.



4. Tap on the template you intend to load, and tap the **Load** button.

## Set up a Low-Flow Test without a Template

1. Tap the **Gear** icon  to access Low-Flow and tap **Launch**.



2. The **Project** screen appears.

iPod 10:15 AM

Default Site Low Flow Criteria

Project

Operator Name: J Smith

Company Name: ISI

Project Name: SF1234

Site Name: Default Site

Turbidity Model: none used

GPS

Latitude 40° 35' 19.75"

Longitude -105° 3' 54.65"

• • • • •

Reports Template Save Next

3. Tap a text-entry field to open the keyboard.
4. Enter the project information. Tap the **Return** button on the keyboard to close the keyboard.
5. Tap the **Next** button to continue to the **Units** screen.

iPod 10:15 AM

Default Site Low Flow Criteria

Units

Well / Tubing: ft / in

Pumping: mL / mL/min

Dissolved O2: mg/L

Conductivity: uS/cm

Temperature: C

• • • • •

Reports Template Save Next

6. If you want to change units, tap the units button and select a different value.
7. Tap the **Next** button to continue to the **Well** screen.

iPod 10:16 AM

Default Site Low Flow Criteria

Well

ID: W3

Diameter: .25 in

Depth to Water est.: 10 ft

Total Depth: 50 ft

Depth Top of Screen: 20 ft

Screen Length: 10 ft

Reports Template Save Next

8. Enter the well information.
9. Tap the **Next** button to continue to the **Stabilization** screen.

iPod 10:17 AM

Default Site Low Flow Criteria

Stabilization

pH: +/- 0.1	pH	<input type="checkbox"/>
Cond: +/- 3	uS/cm	<input checked="" type="checkbox"/>
ORP: +/- 10	mV	<input type="checkbox"/>
Turb: +/- 10	NTU	<input type="checkbox"/>
DO: +/- 0.3	mg/L	<input type="checkbox"/>
Temp: +/- 0.5	C	<input type="checkbox"/>

Reports Template Save Next

10. A green checkmark indicates that the stabilization criterion for the parameter is based on percentage rather than an absolute value. Tap a gray box to change from absolute to percentage.
11. Tap the **Next** button to continue to the **Pump** screen.

iPod 10:25 AM

Default Site **Low Flow Criteria**

**Pump**

Pump Model: P200

Internal Pump Vol.: .5 mL

Tubing Type: Silicone

Tubing Inner Diam.: .25 in

Tubing Length: 6 ft

Pump From TOC: 30 ft

Total System Vol.: 0.148416 L

Reports Template Save Next

12. Enter the pump information. The total system volume is calculated using the internal pump volume, the tubing inner diameter, and the tubing length.
13. Tap the **Next** button to continue to the **Purge** screen.

iPod 10:25 AM

Default Site **Low Flow Criteria**

**Purge**

Pumping Rate: 1 mL/min

☒ Auto Calculate Sample Rate

Sample Rate: 148 min 24 sec

Stabilized Drawdown: 5 in

**Notes:**

Notes will appear in report.

Reports Template Save Start

14. Enter the pumping rate you intend to use during the test.
15. Enter the sampling rate you intend to use, or select **Auto Calculate Sample Rate** if you want the software to assign a sample rate.
16. Enter an estimate of the drawdown you expect during the testing. It is optional to enter notes in the **Notes** field.

- 
17. If you would like to save the test set up information as a template to use again later, tap the **Save** button. You can save it using the default name, or tap the field and enter a new name.

## Install the Pump

1. Determine the static water level.
2. Install the pump in the well.
3. Start the pump and determine the optimum final pumping rate and final stabilized drawdown from static water level. This can be calculated using a graduated cylinder, stopwatch, and water level tape.

## Prepare the Flow Cell



1. Select the barbed NPT fitting that will fit with the tubing size.
2. Tape the threads with plumbing tape.
3. Attach a fitting to the inflow port at base of the cylinder and to the outflow port at the top of the cylinder. Tighten until hand-tight. Do not tighten with a wrench.



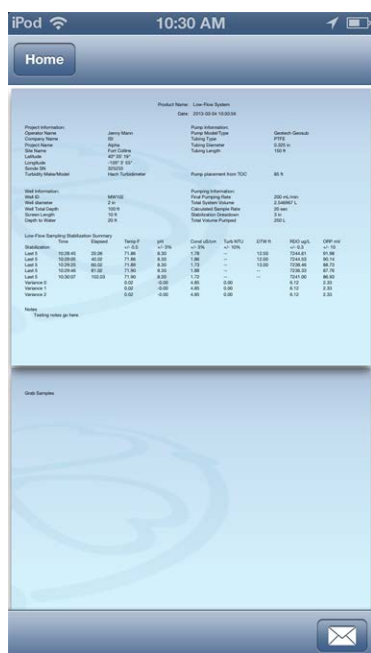
The 3-way valve and check valve are optional.

4. Attach the tubing.
5. Use the attachment screw to connect the flow cell to the base plate.
6. Insert the calibrated SMARTROLL™ MP Instrument.



## Start a Low-Flow Test

1. After you have entered the setup information, installed the pump, and set up the flow cell you are ready to start the Low-Flow test.
2. From the **Purge** screen tap the **Start** button.
3. The test begins and the sample rate countdown is displayed on screen. The parameters appear after the first countdown is complete.
4. When you are satisfied with the stability of the test, tap the **Accept** button.
5. Enter the final values for drawdown, pumping rate, and total volume pumped.
6. You can accept the default file name, or enter a different name. It is optional to enter notes.
7. Click the **Next** button. The report appears. You can pinch and drag the report to resize the view.



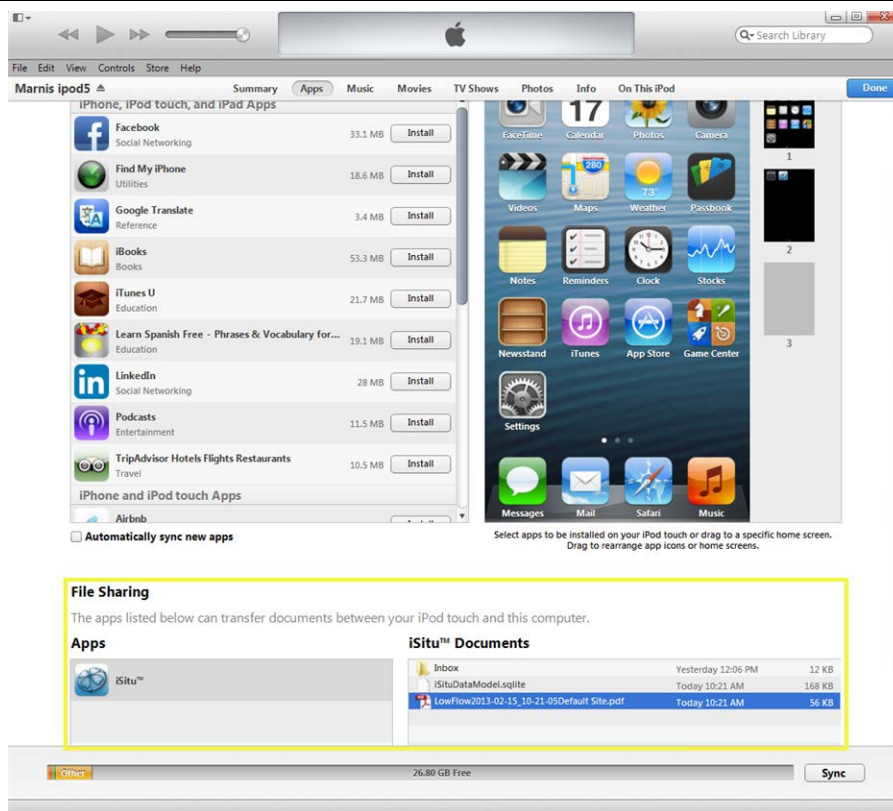
The test report is saved as a PDF file, which can be emailed or transferred to a computer via iTunes®.

## Transfer Low-Flow Report to a Computer

1. Connect the mobile device to a PC with iTunes installed.
2. Click on the Apple device icon next to the eject button.



3. Click the word **Apps** near the top of the screen.
4. Scroll to the bottom of the screen and click on **iSitu**.



5. Click on a file and drag it to your desktop.

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# Care and Maintenance

## Maintenance Schedule

For best results, send the instrument to the manufacturer for factory calibration every 12 to 18 months.

## User-Serviceable Parts

The user-serviceable parts on the instrument include the O-rings, the pH/ORP sensor, and the RDO Sensor Cap.

## O-rings

The instrument has several O-rings that can be maintained by the user in order to keep moisture from entering the instrument and damaging the electronics. Apply a very thin layer of vacuum grease to new O-rings upon installation. The O-rings are located in the following areas.



1	Connector
2	Instrument housing
3	pH sensor
4	RDO Sensor

## RDO Fast Sensor Cap Replacement

The RDO Fast Sensor Cap has a 1-year typical life (15 months of total usage) after the sensor takes its first reading, or 36 months from the date of manufacture. Follow the instructions included in the RDO Sensor Cap Replacement Kit. Replacement caps are available from In-Situ Inc. or your authorized In-Situ distributor.

## pH/ORP Sensor Replacement

To replace the pH/ORP sensor or to refill the reference junction, follow the instructions in the pH/ORP Sensor Instruction Sheet that is included with the replacement sensor.

## Instrument Storage

To store the probe for a week or less, place the probe in the calibration cup with a few drops of clean water to maintain a moist storage environment.

---

To store the probe for more than a week, perform the following procedure.

1. Remove the pH/ORP sensor and place the orange pH port plug into the empty pH/ORP port to prevent any humidity from entering the probe.
2. Locate the sensor storage bottle in which the pH sensor was originally shipped.
3. Open the bottle and remove the O-ring.
4. Moisten the sponge inside the bottle with either a pH storage solution or a pH 4 solution.
5. Slide the O-ring onto the sensor, and then slide the bottle cap over the sensor as shown.



6. Place the sensor tip in the buffer and tighten the cap to prevent the glass bulb from drying.

## **Cleaning the pH/ORP Sensor**

Begin with the gentlest cleaning method and continue to the other methods only if necessary. Do not directly touch or wipe the glass bulb.

To clean the pH sensor, gently rinse with cold water. If further cleaning is required, consider the nature of the debris.

### **Remove Crystalline Deposits**

- Clean the sensor with warm water and mild soap.
- Soak the sensor in 5% HCl solution for 10 to 30 minutes.
- If deposits persist, alternate soaking in 5% HCl and 5% NaOH solutions.

### **Remove Oily or Greasy Residue**

- Clean the sensor with warm water and mild soap.
- Methanol or isopropyl alcohol may be used for short soaking periods, up to 1 hour.
- Do not soak the sensor in strong solvents, such as chlorinated solvents, ethers, or ketones, including acetone.

---

## Remove Protein-Like Material or Slimy Film

- Clean the sensor with warm water and mild soap.
- Soak the sensor in 0.1M HCl solution for 10 minutes and then rinse with deionized water.



Note: After performing any of these cleaning methods, rinse the sensor with water and then soak overnight in pH 4 buffer.

## Cleaning the RDO Sensor

### Clean the Sensor Cap

1. Leave the cap on the sensor.
2. Rinse the sensor with clean water from a squirt bottle or spray bottle.
3. Gently wipe with a soft cloth or brush if biofouling is present.
4. If extensive fouling or mineral build-up is present, soak the RDO Cap end (while the cap is still installed on the sensor) in commercially available household vinegar for 15 minutes, then soak in deionized water for 15 minutes.



Note: Vinegar is safe for all of the sensors on the probe including the RDO Sensor if the Sensor Cap is on.

5. Do not use organic solvents because they will damage the sensing material. Do not remove the cap from the sensor prior to wiping.
6. After cleaning the sensor cap, perform a 2-point calibration.

### Clean the Optical Window

1. Perform this task only once per year when you replace the sensor cap.
2. Pull to remove the sensor cap.
3. Gently wipe the optical window with the supplied lens wipe.



Important: Do not wet the interior lens area with water or any solution.

## Cleaning the Conductivity Sensor

1. Before you begin, ensure that the RDO Cap and the pH/ORP sensor are in place. Rinse the conductivity sensor under running water to remove loose material.
2. Follow Cleaning Procedure 1. If debris is still present, progress to the next cleaning procedure. If the debris is removed, skip to the last step.

---

### **Cleaning Procedure 1**

Avoid damaging the plastic material of the conductivity cell. Gently scrub the conductivity cell with a soft swab and mild soap such as a dilute solution of dish detergent. The probe is shipped with polyurethane foam swabs for this purpose. You can also achieve good results using a gentle back-and-forth motion with a thin cotton pipe cleaner. If debris is still present, continue to Cleaning Procedure 2. If the sensor is clean, skip to the last step.

### **Cleaning Procedure 2**

Avoid damaging the plastic material of the conductivity cell. Gently scrub the conductivity cell with a foam swab and an aggressive soap such as Alconox cleaner. If debris is still present, continue to Cleaning Procedure 3. If the sensor is clean, skip to the last step.

### **Cleaning Procedure 3**

Soak the sensor with dilute acetic acid (10:1 solution) or commercially available household vinegar to pre-soften calcium deposits. Follow this with Cleaning Procedure 1 or Cleaning Procedure 2, depending on the degree of residual contamination. The probe can soak for any length of time in household vinegar. If debris is still present, continue to Cleaning Procedure 4. If the sensor is clean, skip to the last step.

### **Cleaning Procedure 4**

Topically apply dilute phosphoric acid (< 27 %) or the consumer product LIME-A-WAY with a soft swab to remove iron or calcium deposits that remain after using Process 3. Do not allow the cleaner to be in contact with the sensor for more than 10 minutes. Rinse well with clean water and continue to the last step.

Check the sensor calibration before redeployment. Recalibrate the sensor when necessary.

---

## Declaration of Conformity

Manufacturer: In-Situ, Inc.  
221 East Lincoln Avenue  
Fort Collins, CO 80524  
U.S.A.

Declares that the following product:

Product name: smarTROLL™ MP Handheld  
Model: smarTROLL MP  
Product Description: The smarTROLL MP Handheld is a water quality instrument equipped with sensors for measuring dissolved oxygen, conductivity, temperature, pH, ORP, and depth in natural groundwater and surface water. An iOS mobile device running the iSitu App allows instrument control, data display, and data transfer. A battery pack supplies power to the probe and enables wireless communication between the iOS device and the probe. The battery pack includes a barometric pressure sensor and an additional temperature sensor.

The device meets or exceeds the following international requirements and compliance standards:

Under the EMC Directive 2004/108EC

- IEC 61000-6-1: 2005 - Electromagnetic Compatibility (EMC) - Part 6-1: Generic Standards - Immunity for Residential, Commercial and Light-Industrial Environments
- IEC 61000-6-3: 2006 - Electromagnetic Compatibility (EMC) - Part 6-3: Generic Standards - Emission Standard for Residential, Commercial and Light-Industrial Environments
- Electrostatic Discharge Immunity Test (IEC 61000-4-2:2008)
- Radiated, Radio-Frequency, Electromagnetic Field Immunity Test (IEC 61000-4-3:2006, A1:2007, A2:2010)
- EFT/Burst Immunity Test (IEC 61000-4-4:2004, A1:2010)
- Immunity to conducted disturbances, induced by radio-frequency fields (IEC 61000-4-6:2008)
- Power Frequency Magnetic Field Immunity Test (IEC 61000-4-8:2009)
- Radiated Electromagnetic Emissions (CISPR 22: 2008)



Jon Firooz  
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April 5, 2013





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*Instruction Manual*

# Model GK-403

## Vibrating Wire Readout

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# TABLE of CONTENTS

	Page
<b>1. INTRODUCTION .....</b>	<b>1</b>
1.1 FRONT PANEL CONTROLS .....	2
1.2 GETTING STARTED .....	3
<b>2. MODES A-F .....</b>	<b>4</b>
2.1 READINGS DISPLAY .....	4
2.2 READING STORAGE .....	4
2.3 MODES A-F FRONT PANEL CONTROLS .....	5
2.4 SEND DATA .....	6
2.5 CLEAR DATA .....	6
2.6 SET DATE/CLK .....	7
2.7 AUTO INCRMT .....	7
<b>3. MODE G .....</b>	<b>8</b>
3.1 READINGS DISPLAY .....	8
3.2 READING STORAGE .....	9
3.3 MODE G FRONT PANEL CONTROLS .....	10
3.4 SEND DATA .....	11
3.4.1 Row(s) allows the user to transmit sets of readings from all the gages read. Each .....	11
3.4.2 Col(s) allows the user to transmit all the readings from a particular gage read. Each .....	12
3.4.3 All selects all the arrays of data in the readouts Mode G memory. Depending on the .....	12
3.4.4 Factors transmits all the user entered or default calibration Factors, Zero readings, .....	13
3.5 CLEAR DATA .....	13
3.5.1 Row(s) allows the user to clear sets of readings from Mode G memory. Selecting .....	13
3.5.2 Col(s) allows the user to clear all the readings from a particular gage in Mode G .....	14
3.5.3 All clears all the arrays of data in the readouts Mode G memory. Upon selecting the .....	14
3.5.4 RESET will erase all user configurable settings of the GK-403 Vibrating Wire .....	14
3.6 SET DATE/CLK .....	15
3.7 AUTO INCRMT .....	15
3.8 GAGE PARAMS (ALSO SEE APPENDIX E ON APPLICATION NOTES) .....	16
3.8.1 ID allows the user to modify the descriptor for each gage being read. For example, .....	16
3.8.2 Factor allows the user to apply the calibration factor to the sensor being read. The .....	17
3.8.3 Zero allows the user to enter a zero reading for the gage being read. Upon .....	17
3.8.4 Offset allows the user to apply an additional Offset to the measurement being .....	17
3.8.5 Units is a 3 character reference to indicate the engineering units being used. The .....	17
3.8.6 Switch Pos is a most critical GAGE PARAMeter entry. Selection of this option .....	18
<b>4. MAINTENANCE .....</b>	<b>19</b>
4.1 CLEANING: CLEAN THE READER PERIODICALLY WITH A SOFT CLOTH DAMPENED WITH SOAP AND .....	19
4.2 CHARGING: WHEN THE UNIT IS NOT IN USE, ESPECIALLY FOR EXTENDED PERIODS OF TIME, IT .....	19
4.3 CALIBRATION: THE READOUT SHOULD BE SENT PERIODICALLY (EVERY 12 MONTHS) BACK TO THE .....	19
<b>5. TROUBLESHOOTING .....</b>	<b>20</b>
5.1 UNIT WILL NOT COME ON .....	20
5.2 AFTER CHARGING UNIT STAYS POWERED FOR VERY SHORT TIME .....	20
5.3 STORED READINGS ARE NOT RETAINED AT POWER-OFF .....	20
5.4 VIBRATING WIRE GAGE MEASUREMENT SHOWS DASHES .....	20
5.5 VIBRATING WIRE GAGE READING UNSTABLE .....	20
5.6 VIBRATING WIRE GAGE READING IS 9999999 (DISPLAY MODE G) .....	20
5.7 THERMISTOR MEASUREMENT SHOWS DASHES .....	21

5.8 THERMISTOR MEASUREMENT SHOWS 999999.....	21
5.9 READOUT HAS "LOCKED-UP" (READINGS NOT UPDATED, UNRESPONSIVE) .....	21
5.10 CANNOT COMMUNICATE WITH GK-403 VIA RS-232 .....	21
5.11 CONTRAST NEEDS ADJUSTING .....	21
<b>6. LIMITS OF LIABILITY .....</b>	<b>22</b>
<b>APPENDIX A - SPECIFICATIONS .....</b>	<b>23</b>
<b>APPENDIX B - MENU TREES .....</b>	<b>24</b>
<b>APPENDIX C - SAMPLE DATA FILES .....</b>	<b>26</b>
<b>APPENDIX D - ACCESSORIES .....</b>	<b>29</b>
<b>APPENDIX E - APPLICATION NOTES .....</b>	<b>30</b>
<b>APPENDIX F - CONNECTOR PINOUTS .....</b>	<b>32</b>
<b>APPENDIX G - SOFTWARE REPLACEMENT .....</b>	<b>33</b>
<b>APPENDIX H - FILE TRANSFER FROM GK-403 TO IBM PC VIA HYPERTERMINAL.....</b>	<b>34</b>
<b>APPENDIX I - CONFIGURING GK-403 VIA RS-232 .....</b>	<b>38</b>
<b>APPENDIX J - LOAD CELL MODULE.....</b>	<b>39</b>
<b>APPENDIX K - THERMISTOR TEMPERATURE DERIVATION.....</b>	<b>40</b>
<b>APPENDIX L - AUDIO OPTION .....</b>	<b>41</b>

APPENDICES

Page

FIGURE 1 - GK-403 FRONT PANEL .....2

FIGURE 2 - POWER-UP DISPLAY .....3

TABLE 1 - DISPLAY MODE OPTIONS (A-F) .....4

FIGURE 3 - MODES A-F MAIN MENU .....5

FIGURE 4 - MODE G READINGS DISPLAY .....8

FIGURE 5 - MODE G READINGS DISPLAY (NO READINGS) .....8

TABLE 2. 2-DIMENSIONAL STORAGE.....9

FIGURE 6 - MODE G MAIN MENU.....10

FIGURE 7 - MODE G SEND DATA MENU .....11

FIGURE 9 - MODE G PRE--INCREMENT SCREEN .....15

FIGURE 10 - MODE G POST-INCREMENT SCREEN .....16

FIGURE 11 - MODE G GAGE PARAMS .....16

FIGURE 12 - MODE G SWITCH POSITION SCREEN .....18



## **1. INTRODUCTION**

The GK-403 Vibrating Wire Readout provides precise measurements of vibrating wire instruments with increased flexibility over similar instruments. The GK-403 provides complete compatibility with the GK-401 readout, but with many enhancements:

- Larger, multi-line backlit LCD Display
- 5 digit readout of vibrating wire measurements
- Temperature readout (in Celsius units)
- Two modes (A-F and G) of data acquisition and storage
- Internal real-time clock
- Battery-backed clock and memory
- Serial communications port for remote operations and transfer of data to computer
- Easy-to-use control panel

A major feature of the GK-403 is its ability to allow the user to customize nearly every aspect of the instrument's measurement and display functions. The user can:

- a. Enter any values for the parameters used in calculating the vibrating wire reading (calibration factor(k), zero reading, and offset)
- b. Enter an identifier name (up to 10 characters) for each vibrating wire sensor (up to 256)
- c. Enter the units (up to 3 characters) for the vibrating wire readings display
- d. Select various parameters used in exciting the vibrating wire sensor to optimize performance

The GK-403 was designed primarily as an easy-to-use readout instrument. The novice and occasional user will find it very simple to operate. Taking readings, storing new data, and viewing old data are all one keystroke operations.



## 1.1 Front Panel Controls

The front panel of the GK-403 provides the following features:

- a. 15 column x 8 line LCD.
- b. TRANSDUCER connector.
- c. Battery CHARGER connector.
- d. ON/OFF switch.
- e. I/O connector (RS-232 communication port).
- f. 7 position rotary switch (DISPLAY) to select local display mode (A-F are GK-401 compatible).
- g. Joystick, for option selection and cursor movement.
- h. Two push buttons for selection of options and data storage (MENU/ESCAPE and SELECT/STORE).

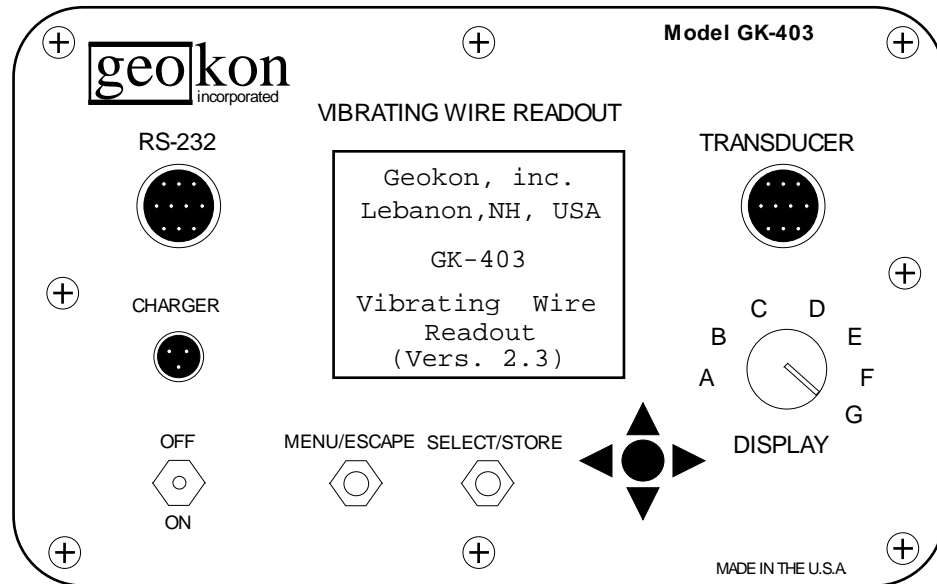


Figure 1 - GK-403 Front Panel

## 1.2 Getting Started

The GK-403 Vibrating Wire Readout should arrive with a patchcord for connecting to the vibrating wire gages. One end will consist of a 10 pin plug for connecting to the respective socket on the faceplate of the GK-403. The other end will consist of 4 or 5 leads terminated with alligator clips. The 4 lead clip is used for convenience when the box is used in areas of low electro-magnetic noise. The 5 lead clip has an additional shield which is connected to the shield wire from the vibrating wire gage cable. Note the colors of the alligator clips are red, black, green, white, and blue. The colors represent the positive vibrating wire gage lead, negative vibrating wire gage lead, positive thermistor lead, negative thermistor lead, and drain wire (shield) lead, respectively. The clips would be connected to their respectively colored leads from the vibrating wire gage cable. In the absence of the blue clip on the 4 clip set the gage cable drain wire (bare) may be connected to the black or green clip.

In the case of connecting the GK-403 leads to direct burial cable (brand Rex) the red lead would connect to the orange wire from the gage, the black lead to the blue wire, the white lead to the white with orange, the green lead to the white with blue, and the shield lead to the aluminum sheath.

Pulling down on the ON/OFF switch turns the unit on. Upon power-up the GK-403 performs several internal systems tests. It checks its battery-backed memory for lost data, the battery voltage, and the real-time clock. While these tests are being done, the LCD displays a title screen (see Figure 2) and the status of the systems test. These tests normally take less than a second to perform, but if any test fails, a warning message will be displayed for several seconds. See Appendix E for descriptions on the possible warning messages and corrective action.

After approximately 1 second, the GK-403 will start taking readings and process and display them based on the setting of the DISPLAY MODE selector. No special action needs to be taken; the GK-403 will continue to make measurements and display the results until automatic power-off caused by;

- a. The user turns the power switch off, or
- b. no front panel activity occurs for 2 minutes.

If the DISPLAY MODE selector is on positions A-F see Section 2 for an explanation of modes A-F. If the DISPLAY MODE selector is on position G see Section 3 for an explanation of mode G.

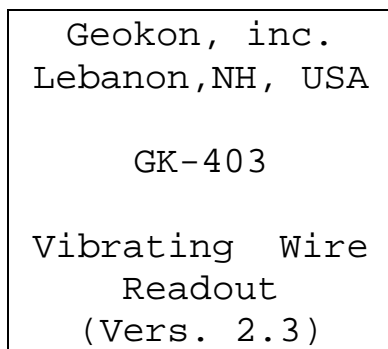


Figure 2 - Power-Up Display

## **2. MODES A-F**

### **2.1 Readings Display**

With the DISPLAY MODE selector in positions A-F the readout will display its vibrating wire and temperature output in large (.6 inch) characters. It will also display the date and time, a REference number (REF,1-256), the DISPLAY MODE selection (SWPOS, A-F), and the default units of the reading.

Each DISPLAY MODE option (A-F) has different preprogrammed parameters which are optimized for certain instruments. Each option also has different scaling and processing characteristics. See Table 1 to determine the best choice for your sensors.

DISPLAY MODE	Use with Geokon Model No.	Calculation	Units	Frequency Sweep (Hz)
A	all	period, $T^*$ in $\mu$ seconds	$\mu$ seconds*	450-6000
B	4300BX,4400, 4500,4600,4700, 4800,4900	$F^2 \times 10^{-3}$	Digits	1200-3500
C	4000	$F^2 \times 10^{-3} \times 4.062$	$\mu$ Strain ( $\epsilon$ )	450-1000
D	4200	$F^2 \times 10^{-3} \times 3.304$	$\mu$ Strain ( $\epsilon$ )	450-1000
E	4100	$F^2 \times 10^{-3} \times 0.39102$	$\mu$ Strain ( $\epsilon$ )	1000-3500
F	4300EX	$F^2 \times 10^{-3}$	Digits	2500-6000

where;

T = Period

F = Frequency in Hertz

\*In DISPLAY MODE position G SWITCH POS A outputs the same as B

Table 1 - DISPLAY MODE options (A-F)

### **2.2 Reading Storage**

Memory for Modes A-F can simply be understood as a single dimensional format. Each vibrating wire gage reading is stored with a REference number to indicate its position in the single dimension. This REference number ranges from 1 to 256, giving a maximum number of readings stored in Modes A-F of 256. Stored with the gage reading and the appropriate REference number are the Julian day, time (24 hour format), temperature, and switch POSition (1-6 representing DISPLAY MODE A-F, respectively).

See Appendix C.1 for a Modes A-F sample data file.

## 2.3 Modes A-F Front Panel Controls

The controls on the front panel take on these functions:

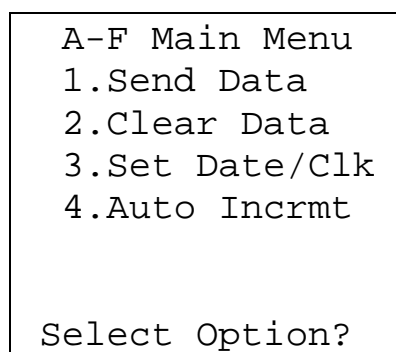
▲ ▼ : Scrolls through the available REference numbers (1 dimensional data storage).

◀ ▶ : Views the data stored with the selected REference number. Shows stored date, time, temperature, vibrating wire reading, and DISPLAY MODE setting when readings were stored. (Blank readings indicate nothing was stored.)

**SELECT/STORE:** Stores the current readings in memory with the currently displayed REference number. Also stores the time, date, and DISPLAY MODE selection. This action will overwrite any previously stored data. The REference indicator may be updated depending on the auto increment selection (Section 2.7).

**MENU/ESCAPE:** Displays main option menu.

The main option menu in modes A-F is as follows:

A rectangular box representing a screen display. Inside the box, the text is as follows:

A-F Main Menu  
1.Send Data  
2.Clear Data  
3.Set Date/Clk  
4.Auto Incrmt  
  
Select Option?

Figure 3 - Modes A-F Main Menu

Pushing ▲ ▼ will advance through these options. When the desired option is reached (indicated by the number being in reverse video) press SELECT/STORE to select. Each selection will be explained in the following sections.

## 2.4 Send Data

Selection of this option will subsequently send all the Modes A-F data currently stored in memory. In Modes A-F there is a possibility of 256 readings. Each reading would include the REference number, day, time, temperature, switch POSition, as well as the actual gage reading (collectively called an array). See Appendix C.1 for a Modes A-F sample data file.

The units of the stored reading would depend on the switch POSition (DISPLAY MODE) when the reading was taken. See Section 2.1 for further information on the units and DISPLAY MODE setting.

The receiving computer should be powered, on-line, and waiting for the data **BEFORE** depressing the SELECT/STORE button with the Send Data option selected. See Appendix H for information on IBM compatible Personal Computer configuration and Hyperterminal to receive files.

Pressing MENU/ESCAPE after selecting Send Data will abort the transmission. Upon transmitting all its Modes A-F data (or aborting) the user will be returned to the Modes A-F Main Menu. Pressing MENU/ESCAPE will return the user to the readings display.

## 2.5 Clear Data

As the name suggests, this option allows the user to clear all of its Modes A-F storage. Press SELECT/STORE to continue with the clearing, or MENU/ESCAPE to abort. **ONLY Modes A-F data will be cleared with this option!** Upon clearing memory (or escaping) the user will be returned to the Modes A-F Main Menu. Pressing MENU/ESCAPE will return the user to the readings display.

## 2.6 Set Date/Clk

Used to set the date and clock information of the GK-403. Normally this option need only be used when first receiving the readout to adjust to local time, to periodically make minor monthly corrections to the date and/or time, or for daylight savings adjustments.

First the user will be asked to modify the date. Scroll through the date components (month:day:year) using ◀ ▶ and adjust them accordingly using ▲ ▼. When finished, press SELECT/STORE to accept and advance to the time entry. Press MENU/ESCAPE to abort the option and return to the Modes A-F Main Menu. Scroll through the time components (hour:minutes) using ◀ ▶ and adjust them accordingly using ▲ ▼. When finished, press SELECT/STORE to accept and return to the Modes A-F Main Menu. Press MENU/ESCAPE to return to the readings display.

## 2.7 Auto Incrmt

This option allows the user to AUTOMatically INCRement the REFERENCE designator of the gage reading. Using ▲ ▼ select option 1 for no increment or 2 for an INCRement of 1 after storing the gage reading. Press SELECT/STORE to accept the selection or MENU/ESCAPE to abort. Upon returning to the Modes A-F Main Menu press MENU/ESCAPE to return to the readings display.

The +1 INCRement is useful where the user is connected to a large terminal box where measurements will be made sequentially on a large number of gages. It can eliminate having to toggle ▲ ▼ after each gage is read to increment the REFERENCE designator.

### **3. MODE G**

#### **3.1 Readings Display**

With the DISPLAY MODE selector in position G, the readout displays the information in normal (small) characters. It shows both the current and stored measurements simultaneously. Screen will look as below:

11/22/91	15:43
NOW	23.7 C
8481.6m/mPOSA	
ROW: 1	COL: 1
ID:1	
11/22/91	15:42
MEM	23.6 C
8481.1m/mPOSA	

Figure 4 - Mode G Readings Display

The top three lines of the display represent the current (NOW) date, time, temperature, vibrating wire reading, and switch POSition. The three characters(m/m) to the right of the vibrating wire gage reading is the default units descriptor. This can be modified by the user for each vibrating wire sensor (1-256). See Section 3.8.6 for additional information on the switch POSition.

ROW and COL indicate the currently active ROW and COLumn in storage memory.

ID indicates the user defined code associated with the active COLumn. Default ID is equal to the COLumn number.

The bottom three lines of the display represent the stored (MEM) date, time, temperature, vibrating wire reading, and switch POSition used at the time of measurement.

If no readings are stored at the current ROW/COLumn setting, the following will display:

11/22/91	15:43
NOW	23.7 C
8481.6m/mPOSA	
ROW: 1	COL: 1
ID:1	
xx/xx/xx	xx:xx
MEM	xxxx
xxxxxxx	POSA

Figure 5 - Mode G Readings Display (no readings)

### 3.2 Reading Storage

Readings in Mode G are stored using a 2-dimensional format. It can be visualized as follows:

		COLumns (1-256)					
ROWs (1-256)		1	2	3	4	5	· · · 256
	1	x	x	x	x	x	· · · x
	2	x	x	x	x	x	· · · x
	3	x	x	x	x	x	· · · x
	4	x	x	x	x	x	· · · x
	5	x	x	x	x	x	· · · x
	·	·	·	·	·	·	·
	·	·	·	·	·	·	·
	·	·	·	·	·	·	·
	256	x	x	x	x	x	· · · x

Table 2. 2-Dimensional Storage

ROWs would represent the readings taken for all the vibrating wire sensors at a particular time. Each gage reading is itself part of an array consisting also of the ROW and COLumn headings, date, time, temperature, and switch POSition. Normally, when the user takes readings in the field on a number of different sensors at the same time, the ROW designation would be used to isolate one set of readings from another. The ROWs value ranges between 1 and 256, allowing the user to store up to 256 sets of readings.

COLumns would represent the readings for a particular vibrating wire gage. Also associated with the COLumn number is a particular gages calibration Factor, Zero reading, Offset, Switch POSition, ID value and Units designator. Changing the COLumn position will apply a new set of values (Factor, Zero, Offset, Switch POSition, ID, Units) to the raw vibrating wire gage reading. Section 3.3 will explain how to change the COLumn setting in the course of use. The COLumns value also ranges between 1 and 256, allowing the user to enter up to 256 sets of sensor information (Factors, Zero, Offset, Switch POSitions, ID's, Units).

COLumn information can be modified using the control panel (Section 3.7) or via the RS-232 port (see Appendix I).

The x's represent the intersection of ROWs and COLumns where readings are stored.



### 3.3 Mode G Front Panel Controls

The controls on the front panel provide the following functions:

▲ ▼ : Increments/Decrements the active ROW (1-256). Any stored data for the ROW (MEM) is shown.

◀ ▶ : Increments/Decrements the active COLUMN (1-256). Any stored data (MEM) is shown.

**SELECT/STORE:** Stores the current reading (at the current ROW/COLUMN settings). The vibrating wire gage reading is stored as well as the temperature, date, and time. This action will overwrite any previously stored data at that ROW/COLUMN.

**MENU/ESCAPE:** Displays Mode G main option menu.

The Mode G main option menu is as follows:

<p>G Main Menu</p> <p>1.Send Data</p> <p>2.Clear Data</p> <p>3.Set Date/Ck</p> <p>4.Auto Incrmt</p> <p>5.Gage Params</p> <p>Select Option?</p>
--

Figure 6 - Mode G Main Menu

Pushing ▲ ▼ will advance through the options. When the desired option is reached (indicated by the number being in reverse video) press SELECT/STORE to select. The Mode G Main Menu is very similar to the Modes A-F Main Menu with the exception of the addition of option 5 (Gage Params). The other options are more involved in Mode G though. Each option will be explained in detail in the following sections.

### 3.4 Send Data

Selecting this option invokes the following menu:

<p>SEND DATA?</p> <p>1. Row(s)</p> <p>2. Col(s)</p> <p>3. All</p> <p>4. Factors</p> <p>Select option?</p>
---

Figure 7 - Mode G Send Data Menu

Following are the sub-option explanations:

#### **3.4.1 Row(s) allows the user to transmit sets of readings from all the gages read.**

**Each** reading in the set would include the ROW and COLUMN designators, day, time, temperature, and switch POSITION. See Appendix C.2 for a Mode G sample data file.

Selecting this option will bring up a screen that informs the user of the START and END ROW. The default STARTing ROW would be the ROW as indicated in the Mode G readings display. To change this default, exit back out to the Mode G readings display (press MENU/ESCAPE thrice) and toggle ▲ ▼ to the appropriate ROW.

The default END ROW matches that of the START, therefore only the current ROW will be transmitted. To modify the END ROW, toggle ▲ ▼. Press SELECT/STORE to transmit based on the START and END ROW parameters.

The receiving computer should be powered, on-line, and waiting for the data **BEFORE** depressing the SELECT/STORE button with the Send Data, Row(s) option selected. See Appendix H for information on IBM or compatible Personal Computer configuration and use of Hyperterminal to receive files.

Pressing MENU/ESCAPE after SELECTing START and END ROWs will abort the transmission. Upon transmitting all its Mode G data (or aborting) the user will be returned to the Mode G Send Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

**3.4.2 Col(s) allows the user to transmit all the readings from a particular gage read. Each** reading in the set would include the ROW and COLumn designators, day, time, temperature, and switch POSition. See Appendix C.2 for a Mode G sample data file.

Selecting this option will bring up a screen that informs the user of the START and END COLumn. The default STARTing COLumn would be the COLumn as indicated in the Mode G readings display. To change this default exit back out to the Mode G readings display (press MENU/ESCAPE thrice) and toggle ◀ ▶ to the appropriate COLumn.

The default END COLumn matches that of the START, therefore only the current COLumn will be transmitted. To modify the END COLumn, toggle ▲ ▼. Press SELECT/STORE to transmit based on the START and END COLumn parameters.

The receiving computer should be powered, on-line, and waiting for the data **BEFORE** depressing the SELECT/STORE button with the Send Data, Col(s) option selected. See Appendix H for information on IBM or compatible Personal Computer configuration and use of Hyperterminal to receive files.

Pressing MENU/ESCAPE after SELECTing START and END COLumns will abort the transmission. Upon transmitting all its Mode G data (or aborting) the user will be returned to the Mode G Send Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

**3.4.3 All selects all the arrays of data in the readouts Mode G memory. Depending on the** number of active ROWs and Columns this option could take some time.

The receiving computer should be powered, on-line, and waiting for the data **BEFORE** depressing the SELECT/STORE button with the Send Data, All option selected. See Appendix H for information on IBM or compatible Personal Computer configuration and use of Hyperterminal to receive files.

Pressing MENU/ESCAPE after SELECTing All will abort the transmission. This option can take a while due to the search that is undertaken to locate the data to transmit. Upon transmitting all its Mode G data (or aborting) the user will be returned to the Mode G Send Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

**3.4.4 Factors transmits all the user entered or default calibration Factors, Zero readings,** Offsets, Switch POSitions, ID values, and Units designators for each active COLUMN in Mode G memory. See Appendix C.3 for a Mode G Factors sample data file. See Appendix H for file transfer instructions. This file is useful in verifying the unique information (calibration Factor, Zero reading, Offset, Switch POSition, ID value, and Units designator) for each gage being read. See Section 3.8 in regard to configuring the gage information.

The receiving computer should be powered, on-line, and waiting for the data **BEFORE** depressing the SELECT/STORE button with the Send Data, Factors option selected. See Appendix H for information on IBM or compatible Personal Computer configuration and use of Hyperterminal to receive files.

Pressing MENU/ESCAPE after SELECTing Factors will abort the transmission. Upon transmitting all its Mode G Factors (or aborting) the user will be returned to the Mode G Send Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

### 3.5 Clear Data

Selecting this option invokes the following menu:

<p>CLEAR DATA?</p> <p>1. Row(s)</p> <p>2. Col(s)</p> <p>3. All</p> <p>4. RESET</p> <p>Select option?</p>
--

Figure 8 - Mode G Clear Data Menu

Following are the sub-option explanations:

#### **3.5.1 Row(s) allows the user to clear sets of readings from Mode G memory.**

**Selecting** this option will bring up a screen that informs the user of the START and END ROW. The default STARTing ROW would be indicated in the Mode G readings display. To change this default exit back out to the Mode G readings display (press MENU/ESCAPE thrice) and toggle ▲ ▼ to the appropriate ROW.

The default END ROW matches that of the START, therefore only the current ROW will be cleared. To modify the END ROW toggle ▲ ▼. Press SELECT/STORE to clear based on the START and END ROW parameters.

Upon clearing the selected ROWs (or aborting by pressing MENU/ESCAPE) the user will be returned to the Mode G Clear Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

**3.5.2 Col(s) allows the user to clear all the readings from a particular gage in Mode G memory.** Selecting this option will bring up a screen that informs the user of the START and END COLUMN. The default STARTing COLUMN would be indicated in the Mode G readings display. To change this default exit back out to the Mode G readings display (press MENU/ESCAPE thrice) and toggle ◀ ▶ to the appropriate COLUMN.

The default END COLUMN matches that of the START, therefore only the current COLUMN will be cleared. To modify the END COLUMN toggle ▲ ▼. Press SELECT/STORE to clear based on the START and END COLUMN parameters.

Upon clearing the selected COLUMNS (or aborting by pressing MENU/ESCAPE) the user will be returned to the Mode G Clear Data menu. Pressing MENU/ESCAPE will return the user to the Mode G Main Menu. Press MENU/ESCAPE again to return to the Mode G readings display.

**3.5.3 All clears all the arrays of data in the readouts Mode G memory. Upon selecting the** user will be asked to verify their intentions by pressing SELECT/STORE. Pressing MENU/ESCAPE will abort the option.

While clearing press MENU/ESCAPE to abort. This option can take a while due to the search that is undertaken to locate the data to clear.

Upon clearing all its Mode G memory (or aborting) the user will be returned to the Clear Data menu. Press MENU/ESCAPE to return to the Mode G Main Menu, press again to return to the Mode G readings display.

**3.5.4 RESET will erase all user configurable settings of the GK-403 Vibrating Wire Readout** (except for Modes A-F data and date and time settings). This includes all Mode G readings, and gage information (calibration Factors, Zero readings, Offsets, Switch POSitions, ID values, and Units designators).

The user will be asked to verify their intentions before the RESET occurs. Pressing SELECT/STORE will proceed with the RESET while MENU/ESCAPE will abort.

Upon clearing all its memory (or aborting) the user will be returned to the Clear Data menu. Press MENU/ESCAPE to return to the Mode G Main Menu, press again to return to the Mode G readings display.

### 3.6 Set Date/Clk

Used to set the date and clock information of the GK-403. Normally, this option need only be used when first receiving the readout to adjust to local time, to periodically make minor monthly corrections to the date and/or time, or for daylight savings adjustments.

First, the user will be asked to modify the date. Scroll through the date components (month:day:year) using ◀ ▶ and adjust them accordingly using ▲ ▼. When through press SELECT/STORE to accept and advance to the time entry. Press MENU/ESCAPE to abort the option and return to the Mode G Main Menu.

Scroll through the time components (hour:minutes) using ◀ ▶ and adjust them accordingly using ▲ ▼. When through press SELECT/STORE to accept and return to the Mode G Main Menu. Press MENU/ESCAPE to return to the readings display.

### 3.7 Auto Incrmt

Two displays are associated with this option. The first refers to the PRE-INCREMENT of the ROW and COLUMN information upon taking a measurement. In other words, the amount the ROW and COLUMN references are changed after storing a measurement. The readings obtained would be stored to the modified ROW and COLUMN references.

The second refers to the POST-INCREMENT of the ROW and COLUMN information. The measurements obtained are stored to the currently displayed ROW and COLUMN references yet, after storing, they are updated according to the selected POST-INCREMENT amounts.

The initial, PRE--INCREMENT screen looks as follows:

PRE--INCREMENT?	
1. (continue)	
BEFORE storage,	
auto-inc ROW?	
2. 0 (default)	
3. +1	4. -1
COL?	5. 0
6. +1	7. -1

Figure 9 - Mode G Pre--Increment Screen

Move the cursor through options 1-7 (using ▲ ▼ and ◀ ▶). To select, press SELECT/STORE. Pressing MENU/ESCAPE returns control to the Mode G Main Menu. Pressing SELECT/STORE advances the user to the POST-INCREMENT option screen as follows:

```

POST-INCREMENT?
1. (continue)
AFTER storage,
auto-inc ROW?
2. 0 (default)
3. +1   4. -1
COL?    5. 0
6. +1   7. -1

```

Figure 10 - Mode G Post-Increment Screen

Move the cursor through options 1-7 (using ▲ ▼ and ◀ ▶), to select press SELECT/STORE. Pressing MENU/ESCAPE returns control to the Mode G Main Menu. Pressing SELECT/STORE the user to the Mode G Main Menu. Press MENU/ESCAPE to return to the Mode G readings display.

### 3.8 Gage Params (Also see Appendix E on Application Notes)

Prompts the user with the following menu:

```

GAGE PARAMS?
1. ID
2. Factor
3. Zero
4. Offset
5. Units
6. Switch Pos
Select Option?

```

Figure 11 - Mode G Gage Params

Each selection is explained as follows;

**3.8.1 ID allows the user to modify the descriptor for each gage being read. For example,** a descriptor could be entered such as "Well #NE8". When the appropriate COLUMN is selected in the Mode G readings display, the ID will display to aid in correlating the COLUMN reference to the gage being read. The COLUMN that the ID will be associated with is selected in the Mode G readings display using ◀ ▶.

The maximum number of characters allowable for the ID is 10. Use ◀ ▶ to move among the 10 positions, then use ▲ ▼ to select the character. The characters available are as follows: lower-case a-z, upper-case A-Z, 0-9, space, question mark, colon, semicolon, left and right angle bracket, equals, ampersand, left and right brackets, apostrophe, underscore, quote, left and right slash, pound, dollar, percentage, asterisk, plus, hyphen, and period.

Press SELECT/STORE to accept the entry, MENU/ESCAPE to abort. Press MENU/ESCAPE to return to Mode G Main Menu, again for Mode G readings display.

### **3.8.2 Factor allows the user to apply the calibration factor to the sensor being read.**

**The** Factor is entered in scientific notation, use ◀ ▶ to move among the digits, ▲ ▼ to change them. BE SURE the exponent is in the correct magnitude and sign. The calibration Factor is normally found on the calibration sheet supplied by Geokon, Inc. for the particular sensor in use. See Appendix E for further information on range and sign conventions.

Press SELECT/STORE to accept, MENU/ESCAPE to abort. Press MENU/ESCAPE twice to return to Mode G readings display.

**3.8.3 Zero allows the user to enter a zero reading for the gage being read. Upon** selecting, the user has the option of using the current reading or entering one. In normal use the user would obtain this reading on the sensor prior to installation, although the Zero reading as found on the calibration sheet can be used.

The Zero reading is entered in scientific notation, use ◀ ▶ to move among the digits, ▲ ▼ to change them. BE SURE the exponent is in the correct magnitude and sign.

Press SELECT/STORE to accept, MENU/ESCAPE to abort. Press MENU/ESCAPE twice to return to Mode G readings display.

**3.8.4 Offset allows the user to apply an additional Offset to the measurement being** made. For example, the elevation of a piezometer could be added to the feet of water being measured above it to output elevation of the water column. The elevation of the piezometer would be entered as the Offset.

The Offset is entered in scientific notation, use ◀ ▶ to move among the digits, ▲ ▼ to change them. BE SURE the exponent is in the correct magnitude and sign.

Press SELECT/STORE to accept, MENU/ESCAPE to abort. Press MENU/ESCAPE twice to return to Mode G readings display.

**3.8.5 Units is a 3 character reference to indicate the engineering units being used.**

**The** default is "m/m". To change use ◀ ▶ to move between the 3 digits, ▲ ▼ to change the selected digits. See the list in Section 3.8.1 of the possible character types.

Press SELECT/STORE to accept, MENU/ESCAPE to abort. Press MENU/ESCAPE twice to return to Mode G readings display.



**3.8.6 Switch Pos is a most critical GAGE PARAMeter entry. Selection of this option brings up the following menu:**

Switch Pos? 1.(A)450-6000 2.(B)1200-3500 3.(C)450-1000 4.(D)450-1000 5.(E)1000-3500 6.(F)2500-6000 Select Option?
--

Figure 12 - Mode G Switch POSition Screen

The user should configure the selected COLumn for the type of gage being read. Select option 1-6 to correspond to the position A-F that would be used if the DISPLAY MODE selected was A-F. See Table 1 for a list of gage model numbers and the appropriate switch POSition. Selecting POSitions C,D,or E, (3,4,or 5) will automatically enter a gage Factor and clear the Zero Reading and Offset entries. See Table 1 regarding the Factor that will be entered.

For example, for a Geokon Model 4500 piezometer select number 2 (DISPLAY MODE position B). The default is position A (1).

The number ranges to the right of the switch position settings (i.e. 450-6000 for A) refer to the frequency range (in Hertz) for that position.

Press SELECT/STORE to accept, MENU/ESCAPE to abort. Press MENU/ESCAPE twice to return to Mode G readings display.

## **4. MAINTENANCE**

The GK-403 Vibrating Wire Readout is designed to operate in field environments, nevertheless there are some basic maintenance procedures that should be followed to insure maximum reliability and functionality. They are as follows:

**4.1 Cleaning: Clean the reader periodically with a soft cloth dampened with soap and water.** DO NOT USE ANY TYPES OF SOLVENTS OR CLEANING AGENTS on the faceplate of the readout. Be careful that no debris of any sort is rubbed on the faceplate as damage to the clear portion covering the LCD may occur.

The connector sockets can be cleaned using a small stiff brush (small painters brush) dipped in soap and water. The sockets are waterproof so the internal electronics will not be adversely affected by them filling with water or other liquids. Be aware however, readings could be affected by shorting or other effects of an improper connection due to moisture present in the connector. Be sure to dry the connectors thoroughly before taking measurements.

**4.2 Charging: When the unit is not in use, especially for extended periods of time, it** should be left connected to the charger. This will ensure a proper charge maintained on the batteries, hence a reduction of the risk of battery failure. Due to the use of Lead-acid batteries in the GK-403, there is little chance of over-charging or memory effects, as is often experienced with Ni-cad type batteries.

**4.3 Calibration: The readout should be sent periodically (every 12 months) back to the** manufacturer for inspection, cleaning, and calibration. A nominal fee will be charged for the service, but it is highly recommended.

## **5. TROUBLESHOOTING**

Listed below are a few commonly experienced problems and remedial action. Contact the factory should a problem arise not explained herein.

### **5.1 Unit will not come on**

Internal battery is probably dead. Charge overnight (8-12 hours). If the unit still fails to come on the fuse may need to be replaced. Follow these steps for fuse replacement;

1. Disassemble according to steps 1-3 of Appendix G.
2. Note fuse under mass-termination connector and inspect for outage (break in filament).
3. Replace fuse if blown.
4. Re-assemble according to steps 8-10 of Appendix G.
5. If unit still fails to power on see Section 5.9.

### **5.2 After charging unit stays powered for very short time**

Internal Lead-acid battery is dead. Consult the factory to schedule battery replacement. Batteries are NOT covered under warranty.

### **5.3 Stored readings are not retained at power-off**

Internal Lithium cell for memory retention is dead. Consult the factory to schedule replacement.

### **5.4 Vibrating wire gage measurement shows dashes**

Check Alligator Clip connections to vibrating wire gage leads. If okay, check gage with Ohmmeter. It should read 50, 90 or 180 ohms depending on the type of gage. (Consult individual spec. sheets). If gage resistance is okay, check Readout with another gage. If still doesn't work consult the factory to schedule return and repair of Readout.

### **5.5 Vibrating wire gage reading unstable**

Improper DISPLAY MODE switch POSition selected. Verify gage model number against Table 1 recommended setting. If switch POSition checks out, gage is marginal or dysfunctional.

### **5.6 Vibrating wire gage reading is 9999999 (DISPLAY MODE G)**

One or more of the constants (Factor, Zero, Offset) are entered incorrectly. This is the overrange indicator. Check all entries. Additional information on appropriate values can be found in Appendix E.

### 5.7 Thermistor measurement shows dashes

Check Alligator Clip connections to Thermistor leads. If okay, check Thermistor with Ohmmeter. It should read between 10K ohms and 2.4K ohms (0 to +30 degrees Celsius). If Thermistor checks out okay consult factory to schedule repair of Readout. Note: Dashes will display if no Thermistor is connected!

### 5.8 Thermistor measurement shows 999999

This is an overrange indication caused by a short of the Thermistor leads. Check the connections to the Thermistor. Check the Thermistor resistance. If zero ohms is indicated (or close to) consult the factory to schedule repair of the transducer.

### 5.9 Readout has "locked-up" (Readings not updated, unresponsive)

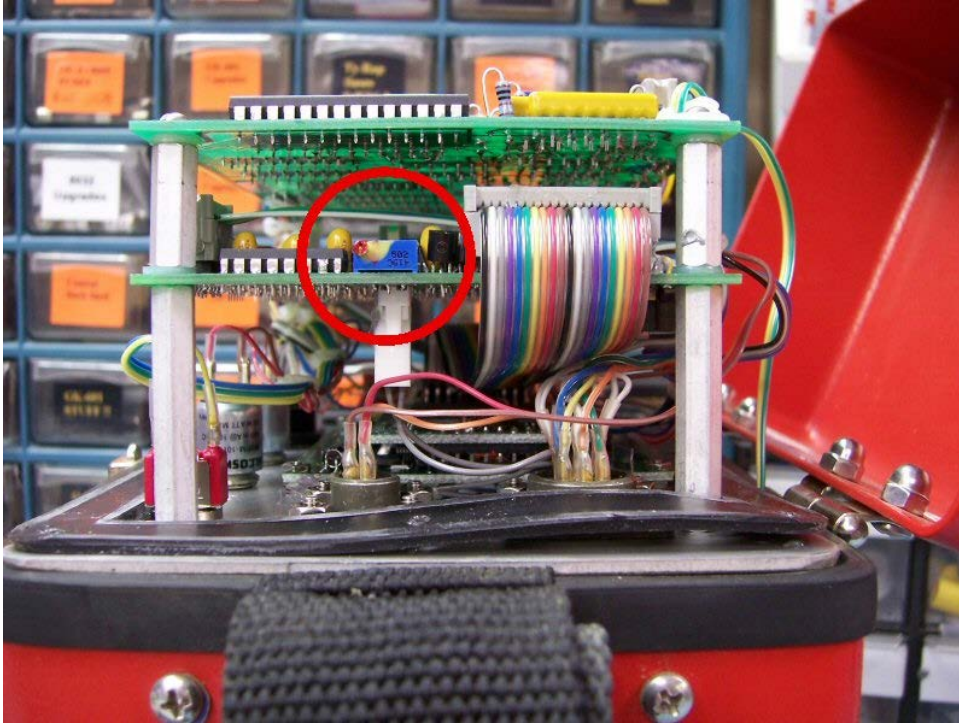
There are many reasons why this condition may arise, for example, the internal Lithium cell may be running low. To reset the unit power it off, then back on. If the Readout still fails to power-up properly then hold both the MENU/ESCAPE and SELECT/STORE buttons down while turning the readout on. **THIS ACTION WILL RESET ALL THE INTERNAL SETTINGS OF THE READOUT**, including the date/time information. **IT SHOULD ONLY BE USED AS A LAST RESORT!**. If the Readout still fails to power-on consult the factory to schedule return and repair of the Readout. Warranty covers all parts (except batteries) and labor for 13 months (12 months plus 1 month grace period) from date of purchase.

### 5.10 Cannot communicate with GK-403 via RS-232

Check communications settings. See Specifications (Appendix A.5) for a list of the proper values. Check the computers COM port with another RS-232 communications device (Modem, Terminal, PC, etc.). If all else fails contact the factory for assistance.

### 5.11 Contrast needs adjusting

Refer to the photo on the next page to locate Trimpot R13 on the CPU circuit board. Turn clockwise to lighten the display, counter-clockwise to darken.



## **6. LIMITS OF LIABILITY**

The GK-403 Vibrating Wire Readout has been developed specifically for use with Geokon, Inc. vibrating wire gages and, as such, Geokon, Inc. assumes no responsibility for its use with other systems. Every effort has been made to ensure reliable operation, but the user must be aware that there is no warranty against uninterrupted or trouble-free operation. For the user conducting particularly unusual or sensitive analysis, or for those not familiar with vibrating wire gage data processing, it is recommended that the problems be double checked using another measurement system.

Also, the readout is provided 'as is' and Geokon, Inc. assumes no responsibility as to results, performance, or interpretation associated with the GK-403 Vibrating Wire Readout. Warranty shall cover parts (except batteries) and labor for a period of 1 year from the date of purchase. In addition, there is a 1 month grace period to the warranty for a total of 13 months.

We reserve the right to revise this publication and/or readout from time to time with no obligation to notify users of these changes.

**All things considered, Geokon, Inc., is not liable for any claims, injuries, or damages caused directly or indirectly by the proper or improper use of the GK-403 Vibrating Wire Readout, beyond the purchase price of the Readout.**

## **APPENDIX A - SPECIFICATIONS**

### **A.1 Vibrating Wire Readout:**

Excitation Range: 400 Hz to 6000 Hz, 5 volt square wave  
Measurement Resolution: 0.1 $\mu$ s  
Time base Accuracy:  $\pm$  50ppm

### **A.2 Temperature Readout:**

Sensor Type: Thermistor, Dale #1C3001-B3 (YSI 44005)  
Sensor Accuracy:  $\pm$ 0.5° Celsius  
Measurement Range: -50° to +150° Celsius  
Measurement Resolution: 0.1° Celsius  
Measurement Accuracy: 0.5% to 1.0% FSR

### **A.3 Memory:**

RAM: 64K Static, 48K Used  
ROM: 32K EPROM, 16K Used  
Reading Storage: 2000 arrays  
Array Partition: 256 arrays for Modes A-F, remaining for Mode G

### **A.4 Real-Time Clock:**

Features: Full calendar with automatic leap year correction  
Time Format: 24 hour (hhmm)  
Date Storage Format: Julian Day  
Date Display Format: month/day/year (mm/dd/yy)  
Oscillator: 32.768 kHz  
Accuracy:  $\pm$ 1 minute per month

### **A.5 Communications:**

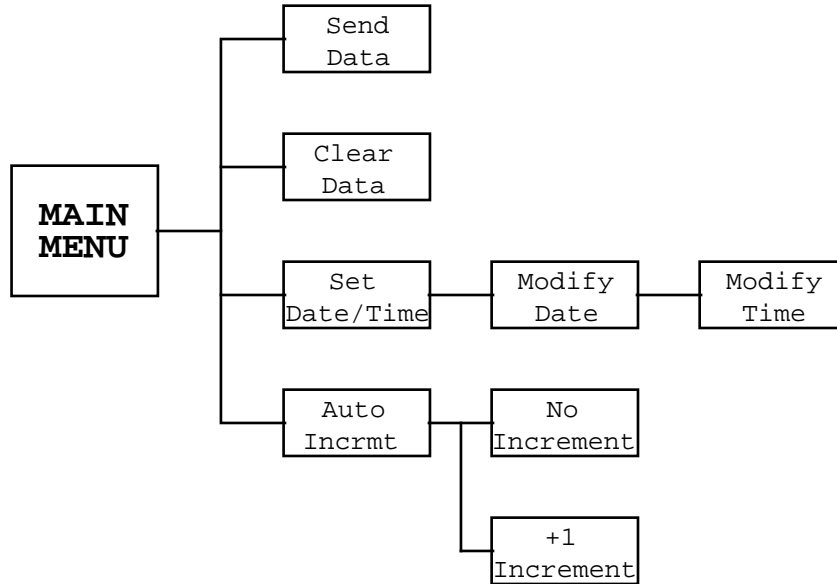
Parameters: 9600 baud, 8 data bits, 1 stop bit, no parity, full duplex, user configurable  
Handshake: XON/XOFF  
Transmission Format: ASCII

### **A.4 Physical:**

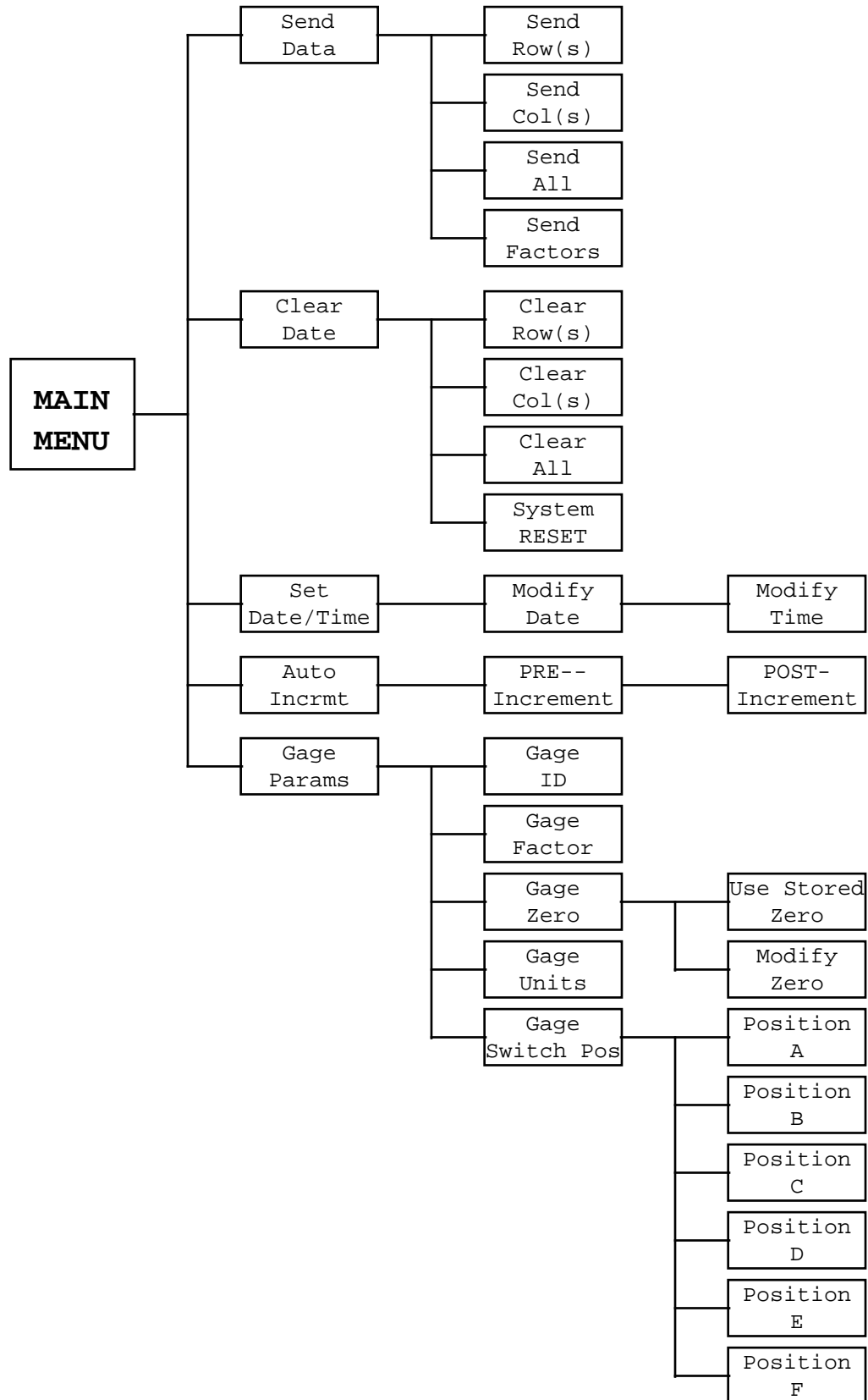
Display: 15 Column x 8 Line Backlit LCD  
Dimensions: 19.1cm (7.5") x 13.3cm (5.25") x 23.5cm (9.25")  
Weight: 2.7kg (6 lbs.)  
Temperature Range: -10° to +50° Celsius  
Battery: 12 volt, 3.4 AHr (Powersonic PS-1230)  
Operating Time: approx. 10 hours

## **APPENDIX B - MENU TREES**

### **B.1 Modes A-F Menu Tree**



## B.2 Mode G Menu Tree





## **APPENDIX C - SAMPLE DATA FILES**

All data files are comma delineated ASCII format. In other words, the data points in each array are separated by commas.

### **C.1 Modes A-F Data File**

```

1,326,1345,8480.2,21.3,2
2,326,1345,8480.1,21.3,2
3,326,1345,8480.0,21.3,2
4,326,1345,8480.2,21.3,2
5,326,1345,8480.5,21.3,2
6,326,1345,8479.6,21.3,2
7,326,1345,8480.4,21.3,2
8,326,1345,8480.1,21.3,2
9,326,1345,8479.9,21.3,2
10,326,1345,8480.0,21.3,2
11,326,1345,8480.1,21.3,2
12,326,1345,8480.2,21.3,2
13,326,1345,8480.1,21.3,2
14,326,1345,8480.2,21.3,2
15,326,1345,8480.1,21.3,2
16,326,1345,8480.2,21.3,2
17,326,1345,8479.7,21.3,2
18,326,1345,8479.8,21.3,2
19,326,1345,8480.3,21.3,2
20,326,1345,8479.9,21.3,2
21,326,1345,8479.8,21.3,2
22,326,1345,8480.1,21.3,2
23,326,1345,8480.0,21.3,2
24,326,1345,8479.9,21.3,2
25,326,1345,8480.1,21.3,2
26,326,1346,8480.2,21.3,2
27,326,1346,8480.0,21.3,2
28,326,1346,8479.9,21.3,2
29,326,1346,8479.6,21.3,2
30,326,1346,8479.7,21.3,2

```

where;

Column 1 represents the REference number (1-256)

Column 2 represents the Julian Day

Column 3 represents the Real Time (24 Hr. format)

Column 4 represents the vibrating wire gage reading

Column 5 represents the temperature (degrees Celsius)

Column 6 represents the switch POSition (1-6 for A-F, respectively)

## C.2 Mode G Data File

```

1, 1,331,1030, 8472.90,24.7,2
1, 2,331,1032, 9894.70,24.7,2
2, 1,331,1031, 8473.00,24.7,2
2, 2,331,1032, 9893.70,24.7,2
3, 1,331,1031, 8472.90,24.7,2
3, 2,331,1032, 9894.50,24.7,2
4, 1,331,1031, 8472.70,24.7,2
4, 2,331,1032, 9895.10,24.7,2
5, 1,331,1031, 8472.70,24.7,2
5, 2,331,1032, 9895.40,24.7,2
6, 1,331,1031, 8473.30,24.7,2
6, 2,331,1032, 9895.00,24.7,2
7, 1,331,1031, 8473.30,24.7,2
7, 2,331,1032, 9895.00,24.7,2
8, 1,331,1031, 8473.30,24.7,2
8, 2,331,1032, 9895.30,24.7,2
9, 1,331,1031, 8473.30,24.7,2
9, 2,331,1032, 9895.20,24.7,2
10, 1,331,1031, 8473.30,24.7,2
10, 2,331,1032, 9895.20,24.7,2
11, 1,331,1031, 8472.80,24.7,2
11, 2,331,1032, 9895.20,24.7,2
12, 1,331,1031, 8472.80,24.7,2
12, 2,331,1032, 9895.20,24.7,2
13, 1,331,1031, 8472.80,24.7,2
13, 2,331,1032, 9895.20,24.7,2
14, 1,331,1031, 8472.80,24.7,2
14, 2,331,1032, 9895.60,24.7,2
15, 1,331,1031, 8472.80,24.7,2
15, 2,331,1032, 9895.10,24.7,2

```

where;

Column 1 represents the ROW (1-256)

Column 2 represents the COLumn (1-256)

Column 3 represents the Julian Day

Column 4 represents the Real Time (24 Hr. format)

Column 5 represents the vibrating wire gage reading

Column 6 represents the temperature (degrees Celsius)

Column 7 represents the switch POSition

### C.3 Mode G Factors File

```

1, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,1
2, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,2
3, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,3
4, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,4
5, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,5
6, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,6
7, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,7
8, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,8
9, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,9
10, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,10
11, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,11
12, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,12
13, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,13
14, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,14
15, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,15
16, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,16
17, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,17
18, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,18
19, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,19
20, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,20
.      .      .      .      .      .
.      .      .      .      .      .
.      .      .      .      .      .
254, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,254
255, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,255
256, 1.000000E+00, 0.000000E 00, 0.000000E 00,1,m/m,256

```

where;

Column 1 represents the COLumn number (1-256)

Column 2 represents the Factor (scientific notation)

Column 3 represents the Zero (scientific notation)

Column 4 represents the Offset (scientific notation)

Column 5 represents the switch POSition (1-6 for A-F, respectively)

Column 6 represents the Units (up to 3 ASCII characters)

Column 7 represents the ID (up to 10 ASCII characters)

## **APPENDIX D - ACCESSORIES**

The following accessories arrive with the GK-403 Vibrating Wire Readout:

1. 100-240VAC 50/60 Hz 18VDC Power Supply.
2. Vibrating wire gage interconnect cable (flying leads).
3. 9 Pin RS-232 communications cable.
4. USB-to-Serial adapter cable.
5. GK-403 Vibrating Wire Readout manual.
6. Spare 1A fuses (5).
7. Geokon Terminal Window Software

The following are optional accessories:

1. Load Cell Module (for connecting to Vibrating Wire Load Cell).
2. Model 8032 16 or 32 channel Multiplexer.
3. GK-403-4 Audio Option.

## **APPENDIX E - APPLICATION NOTES**

### **E.1 Mode G Equation and Display Units**

For vibrating wire transducers, the output frequency of the gage responds according to the following equation:

With a negative Factor;

$$\text{Output} = \text{Factor} \times (\text{Zero Reading} - \text{Current Reading}) + \text{Offset}$$

With a positive Factor;

$$\text{Output} = \text{Factor} \times (\text{Current Reading} - \text{Zero Reading}) + \text{Offset}$$

where;

**Output** is Psi, force, ft water, etc.

**Factor** translates to the new units

**Zero Reading** is the vibrating wire frequency squared reading ( $\times 10^{-3}$ ) at the zero condition

**Current Reading** is the vibrating wire frequency squared reading ( $\times 10^{-3}$ ) at the moment

**Offset** scales the reading accordingly

Note that for gages under compression, the frequency of oscillation of the gage decreases with increasing force. See Table E-1 for the appropriate sign given the gage model number.

Geokon Model	Description	Factor Sign
4000	Strain Gage	+
4100/4150	Strain Gages	+
4200/4210	Strain Gages	+
4300BX	BX Borehole Stressmeter	+
4300EX	EX Borehole Stressmeter	+
4300NX	NX Borehole Stressmeter	+
4350	Biaxial Stressmeter	+
4360	Stress Ring	+
4400	Embedment Jointmeter	+
4420	Crackmeter	+
4450	Displacement Transducer	+
4500	Piezometer	-
4600/4651/4675	Settlement Systems	+/-
4700	Temperature Sensor	+
4800/4850	Pressure Cells	-
4900	Load Cell	-
4910/4911/4912	Load Bolts	+

Table E-1 - Vibrating Wire Gage Factor Signs

## E.2 Mode G Decimal Point Placement

The decimal point placement of the gage reading is decided by the position of the exponent in the Factor. For example, a Factor of +1.0 will cause a reading of  $\pm 12345.6$ , with leading zeros and the plus sign suppressed. Possible decimal point positions range from  $\pm 0.123456$  to  $\pm 123456$ . Equivalent Factor exponents are from  $1 \times 10^{-5}$  to  $1 \times 10^1$ .

## E.3 Terminal Emulation with GK-403

The various Gage Parameters the GK-403 retains in its Mode G memory (Factor, Zero Reading, Offset, etc.) can be altered 3 ways. As has been discussed in Section 3.8, values can be entered via the front panel controls. Secondly, Appendix I details entering these parameters by creating a file on the PC and downloading it to the Readout. A third method will be discussed in this section, namely; changing or entering values in a terminal emulation mode with the GK-403.

The format for entering Factors, Zero Readings, etc., is as follows:

**COLumn,Factor,Zero Reading,Offset,Switch POSition,Units,ID <CR>**

For example, type: **3,.001,7034,35,2,Psi,MAINPUMP <CR>**

The above example selected COLumn 3 gage, entered a Factor of .001, Zero Reading of 7034, Offset in Psi of 35, Units of Psi, and ID of MAINPUMP.

The proper entry of these values can be verified by typing a question mark and the COLumn number to check. For example, **?3 <CR>** will return "3,.001,7034,35,2,Psi,MAINPUMP".

The format is comma-delineated so to change one value commas can be supplied to skip values NOT to be changed. For example, **3,,,40 <CR>** will enter a new Offset of 40. No other parameters will be changed. Type **?3 <CR>** to verify the change.

Another example, **3,,,,,OLDPUMP <CR>** will change only the ID. Type **?3 <CR>** to verify.

When through changing values, disconnect the RS-232 cable interface. The values entered will be retained in the Readout at power-off.

## **APPENDIX F - CONNECTOR PINOUTS**

### **F.1 Transducer Plug Pinout**

10-pin Bendix Plug (PT06F-12-10P)	Wire Color	Alligator Clip Boot Color (Flying Leads)	Description
A	Red	Red	VW Gage +
B	Black	Black	VW Gage -
C	White	White	Thermistor +
D	Green	Green	Thermistor -
E	Bare	Blue	Shield
F			+12 VDC Power
G			Power Ground
H			Mux Sense
J			Mux Clock
K			Mux Type

### **F.2 Charger Pinout**

3-pin Bendix Plug (PT06E-8-3P)	Wire Color	Description
A	Red	System Ground
B	Brown	VWG+
C	Orange	Charger Input

### **F.3 RS-232 Plug Pinout**

10-pin Bendix Plug (PT06F-12-10P)	Wire Color	DB-9 RS-232	DB-25 RS-232	Description
A	Brown	5	7	Ground
B	Red	3	2	TXD
C	Orange	2	3	RXD
D	Yellow	7	4	RTS
E	Green	8	5	CTS
F	Blue			Ground
G	Violet	4	20	DTR
H				
J	White			Ground
K	Black			+12 VDC

## **APPENDIX G - SOFTWARE REPLACEMENT**

### **Instructions:**

1. Remove the 8 screws located on the perimeter of the GK-403 faceplate.
2. Remove the faceplate and electronics assembly by pulling on the selector knob. Gently lift the assembly from the enclosure so that no wires catch on the faceplate brackets and are damaged.
3. Disconnect the 2 and 3 pin Molex connectors between the battery pack and LCD backlighting power supply and electronics assembly.
4. Turn electronics assembly so that the front of the faceplate faces away. Note 4 screws securing printed circuit board (PCB) to assembly. Remove screws.
5. Disconnect the two 16-pin mass termination connectors located near the Serial Number designation on the component side of the bottom-most PCB. Disconnect the 16-pin mass termination connector located near the fuse on the component side of the same PCB.
6. Lift the bottom-most PCB from the electronics assembly. Note the existing EPROM (part 27C256) with the label "GK-403 MAIN" on it. Note orientation of notch on one end of the installed EPROM. Gently pry either side of the EPROM with the small regular screwdriver until it is free from its socket.
7. Insert new EPROM with same orientation as former into socket. Before pushing gently into the socket make sure ALL the pins on the new EPROM line up with their respective holes in the socket!
8. Re-connect mass termination connectors, re-affix PCB onto assembly, re-connect MOLEX connectors, and re-insert electronics assembly in the enclosure. Make sure when lowering assembly into the enclosure that no wires are pinched against the faceplate brackets!
9. Fasten faceplate and electronics assembly into enclosure using the 8 Phillips head screws.
10. EPROM replacement complete.

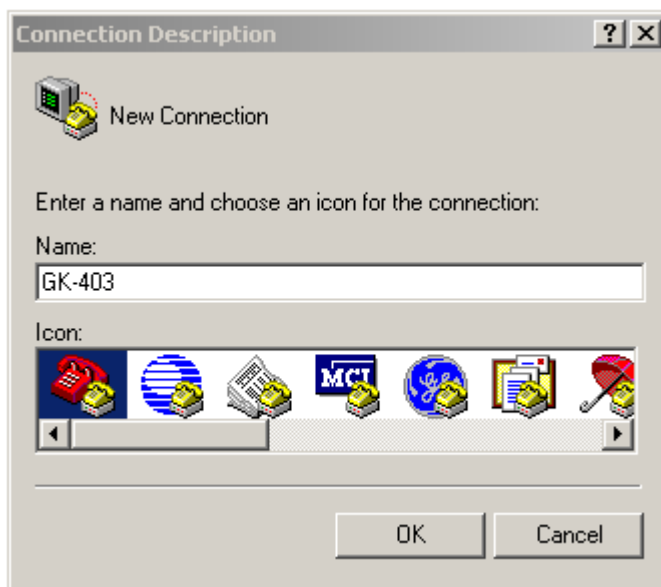


## **APPENDIX H - FILE TRANSFER FROM GK-403 TO IBM PC VIA HYPERTERMINAL**

***NOTE: EFFECTIVE APRIL 2013, GEOKON'S TERMINAL WINDOW SOFTWARE(GEO-TWS) IS INCLUDED WITH NEW GK-403 SHIPMENTS. SHOULD THE COMPUTER USED FOR FILE TRANSFER OPERATE USING WINDOWS VISTA/ W7 / W8, THESE OPERATING SYSTEMS DO NOT HAVE THE HYPERTERMINAL PROGRAM PRE-INSTALLED AS PREVIOUS VERSIONS OF THE WINDOWS OS DID. PLEASE INSTALL THE GEOKON TERMINAL WINDOW SOFTWARE AND READ THE ON-LINE HELP SECTIONS FOR USING THIS SOFTWARE TO TRANSFER DATA. THE SOFTWARE IS ALSO AVAILABLE FOR DOWNLOAD AT: [www.geokon.com/software](http://www.geokon.com/software)***

Start Hyperterminal: Start | Programs | Accessories | Communications

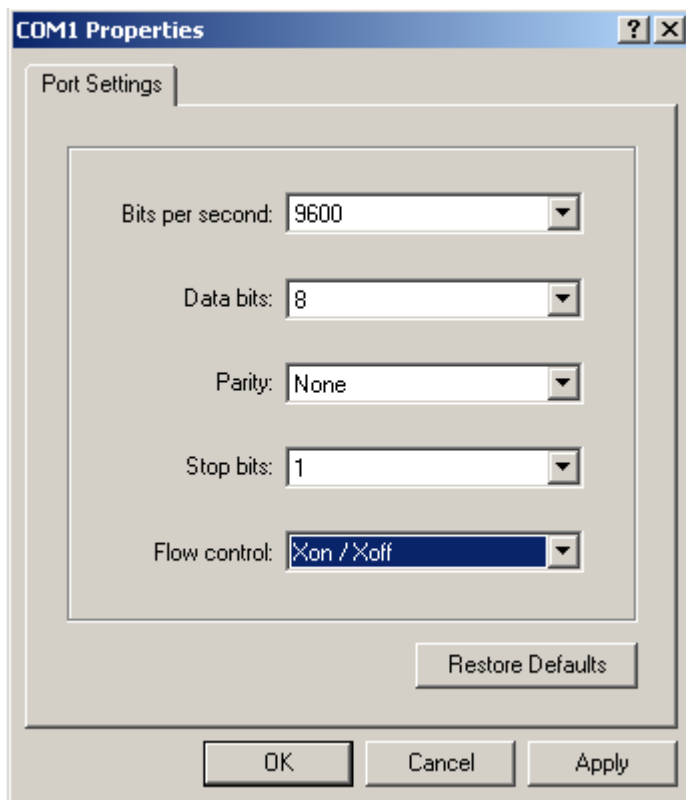
Enter a name for the Connection. Select OK.



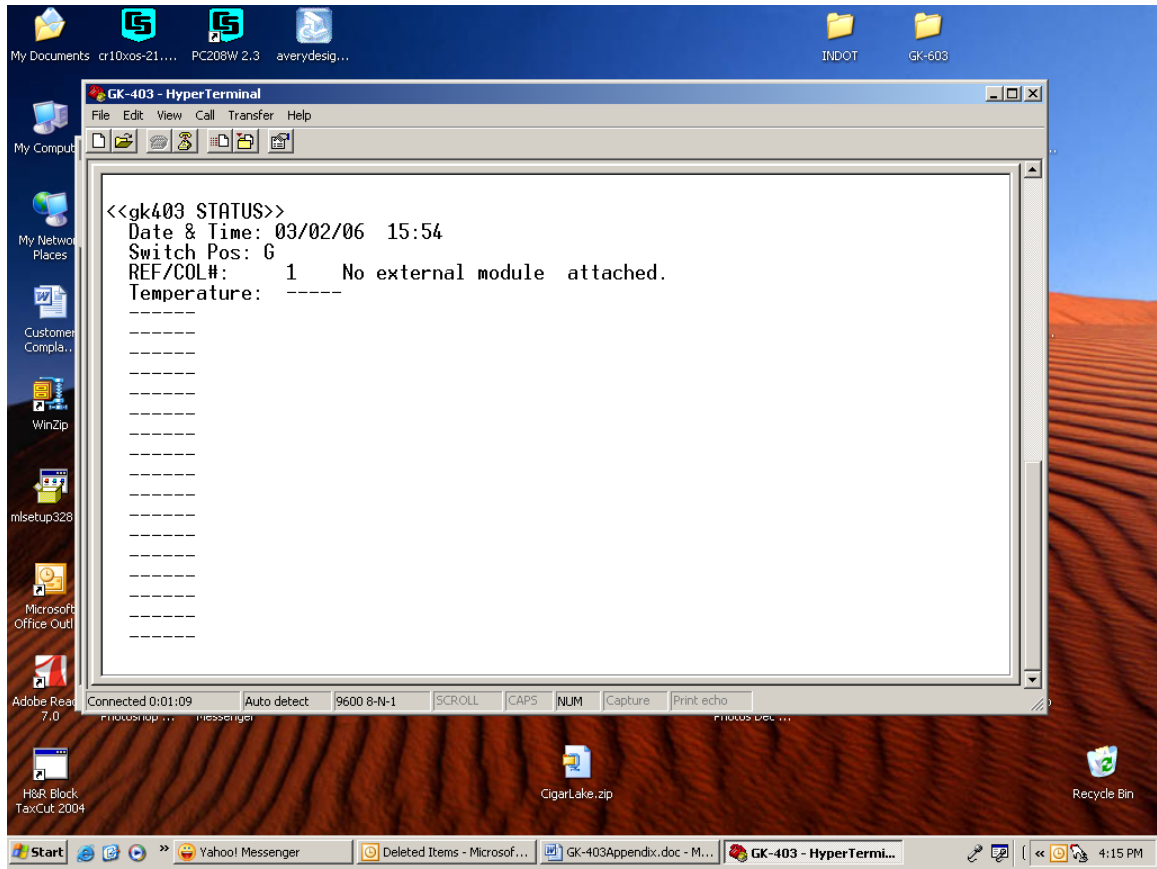
Change the Connect Using setting to the appropriate COM port for the computer being used. Select OK.



Enter the Port Settings as shown. Select Apply. Select OK.

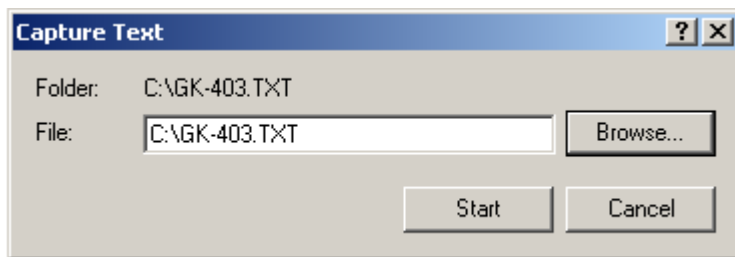


With the RS232 cable connected to the RS232 port on the GK-403 and the unit turned on and in DISPLAY position G put the cursor in the Hyperterminal display screen and push the Enter key a few times to verify communications has been established. The text as shown below should appear.

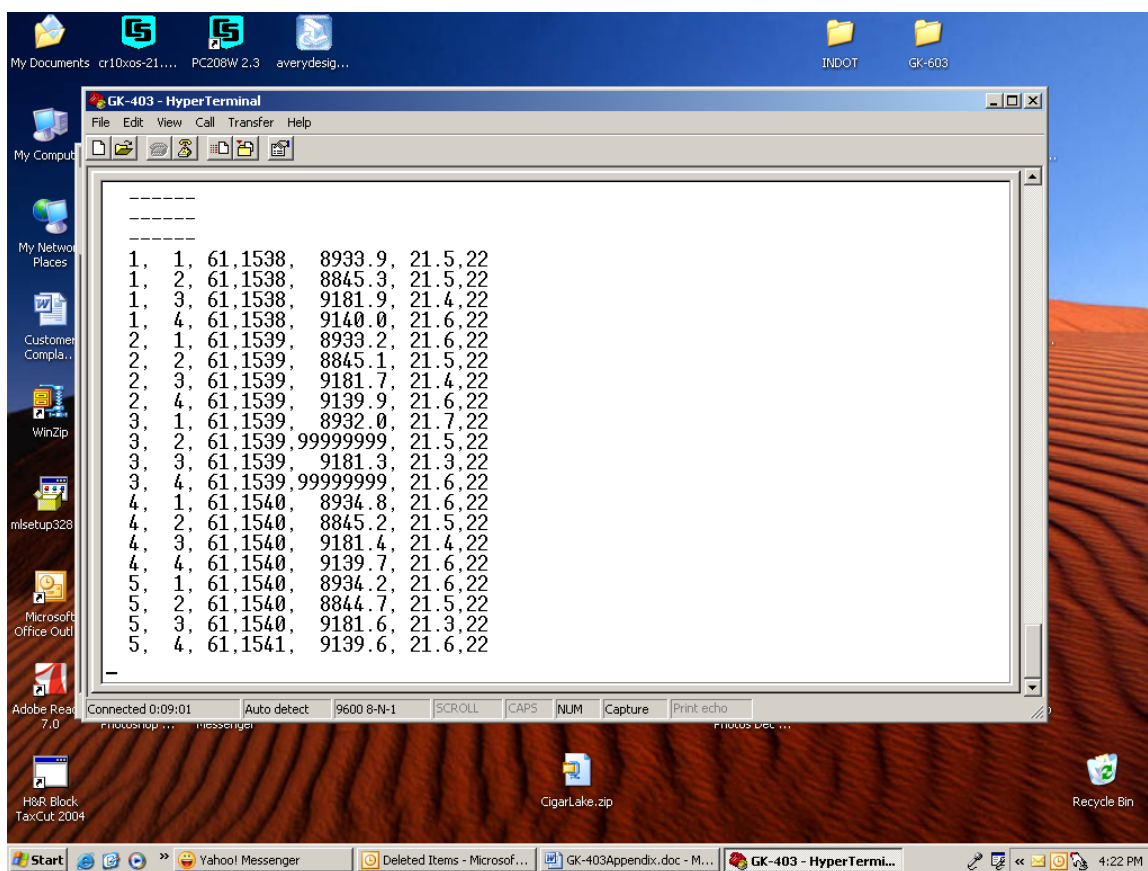


Upon confirmation of communications select Transfer | Capture Text.

Enter the Path and name of the file you wish to create, either directly or with the Browse button. Select Start.



The display of the GK-403 should be set to position G. Push the Menu/Escape button and put the toggle on No. 1 Send Data. Push the Select/Store button. Depending on what information is required (Row (s), Col(s) All, or Factors) move the cursor to the appropriate selection. Push Select/Store. The data should scroll across the screen as shown below.



The download is now complete. A text file now exists as specified by the User. You can open this file in NotePad or WordPad. It can also be opened in Excel.

Push the Menu/Escape button twice to take you back to the main display mode. Exit Hyperterminal and save the settings as appropriate.

**APPENDIX I - CONFIGURING GK-403 VIA RS-232**

All unique gage information (calibration Factors, Zero readings, Offsets, Switch POSitions, ID values, and Units) used in Mode G of the Readout can be downloaded to the reader to save having to enter manually. This file can be created on the PC using an editor (see Appendix C.3 for details on the file format) or uploaded from the GK-403 after entering the appropriate values manually. After uploading, the file can be modified as need be, then downloaded back into the GK-403. See Appendix C.3 regarding details of the Factors file format.

**APPENDIX J - LOAD CELL MODULE**

**Refer to the Model 4900 VW Load Cell Manual**



## **APPENDIX L - AUDIO OPTION**

### **L.1 Introduction**

The audio option, when connected to the GK-401 or GK-403 Readout Box is a useful tool for assessing the quality of a vibrating wire sensor installation. It is roughly the equivalent of having an oscilloscope connected to the readout box, when, instead of a visual trace of the frequency output, there is an audible tone produced in a head set worn by the installer. The audio option can be useful in diagnosing the reasons for poor gage performance and in selecting a remedy.

### **L. 2 Construction**

The audio option consists of a headset connected to an audio amplifier/battery pack which plugs into the charger socket of the face plate of the readout box.

### **L. 3 Application**

Good installations are characterized by a clear strong audible tone which decays slowly. The shorter the length of the sensor vibrating wire and the greater its tension the higher is the tone or pitch of the output signal. In general, the ringing of the gage can be heard to continue from one excitation pulse to the next.

Gages in which the output signal decays rapidly due to a dampening of the natural frequency of vibration of the gage wire may have dirt or moisture on the wire. A temporary fix, in emergencies, might be to dislodge the dirt or moisture by knocking on the gage.

In some cases, the body of the gage vibrates at a resonant frequency which dampens the natural frequency of the wire itself. These effects often disappear as soon as the body or ends of the gage are properly restrained. This sometimes happens where concrete embedment gages are held in place using iron wire: as soon as the concrete is poured, the output signals become clear and strong. This problem can often be remedied by altering the tension of the iron wire, or by using a more rigid method of holding the gages in place prior to pouring the concrete.

In rare instances, resonance of the gage body interferes with the output signal over a very narrow band of frequencies. On either side of this frequency band, the signal is clear and strong. Sometimes raising or lowering the transducer relative to the water level will eliminate the problem.

Gages may emit an unsteady tone due to the presence of noise or harmonics. The general effect is to cause unsteadiness in the readout digits.

Noise can sometimes be caused by the proximity of power lines and electrical machinery. It can often be removed by connecting the sensor shield to the ground wire



clip on the readout box or by shielding the transducer itself. Harmonics are most frequently encountered in longer sensors (strain gages) where the longer wire is at a low tension. In these cases it may be possible to remove the harmonics by plucking the gage at a lower frequency or by retensioning the wire.

#### L. 4 Weak gages

Weak gages give an output signal with a low amplitude. Perhaps the gage is being plucked at the wrong frequency in which case the signal might be improved by switching to another DISPLAY position ( 403 readout box) or by adjusting the pluck frequencies on a datalogger. With strain gages using detachable coils, the coil may have slipped and needs repositioning. By using the audio option, it is possible to find the optimum position for the coil.

Weak gages may also be the result of cable damage, in which case repair or replacement of the cable or its connector could solve the problem.



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*Instruction Manual*  
**Model LC-2**  
Single Channel Datalogger



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# TABLE OF CONTENTS

<i>Instruction Manual</i> .....	<i>1</i>
MODEL LC-2 .....	1
SINGLE CHANNEL DATALOGGER.....	1
<b>1. INTRODUCTION .....</b>	<b>1</b>
<b>2. LC-2 MODEL TYPES.....</b>	<b>1</b>
2.1 RS-232 INTERFACE (8002-1-1, 8002-1A-1):.....	1
2.2 USB INTERFACE (8002-1-2, 8002-1A-2): .....	1
2.3 RS-485 INTERFACE (8002-1-3, 8002-1A-3):.....	2
<b>3. GETTING STARTED.....</b>	<b>3</b>
3.1. TRANSDUCER INSTALLATION:.....	3
3.2. SOFTWARE INSTALLATION AND SETUP .....	5
3.2.1. <i>LogView</i> Installation:.....	5
3.2.2. <i>Launching LogView</i> :.....	5
3.2.3 <i>LogView</i> Workspaces:.....	6
3.2.4 <i>Adding Projects to LogView Workspaces</i> :.....	7
3.2.5 <i>Adding Dataloggers to LogView Projects</i> : .....	8
3.2.6. <i>LC-2 Connection (8002-1-1, 8002-1A-1)</i> :.....	10
3.2.7. <i>LC-2 Connection (8002-1-2, 8002-1A-2)</i> :.....	10
3.2.8. <i>Connecting to a Datalogger with LogView</i> :.....	11
3.2.9. <i>Determining COM Port Numbers</i> :.....	12
3.3. EXAMPLE SETUP USING TEXT COMANDS.....	14
<b>4. COMMAND LIST .....</b>	<b>20</b>
4.1.    BRNNN .....	21
4.2.    C .....	21
4.3.    CSMM/DD/YY/HH:MM:SS .....	21
4.4.    DEFAULT .....	22
4.5.    DF .....	23
4.6.    DNNNN .....	23
4.7.    E.....	24
4.8.    GNN/SZZZZ/SM MMM/SOOOO OR GNN/SAAAA/SBBBB/SCCCC.....	25
4.9.    IDDDDDDDDDDDDDDDDD .....	28
4.10.   LC .....	28
4.11.   L.....	28
4.12.   LN/LLLL/III .....	29
4.13.   LD .....	30
4.14.   LE .....	30
4.15.   M.....	30
4.16.   MD.....	30
4.17.   ME.....	30
4.18.   MS .....	31
4.19.   N .....	31
4.20.   NA .....	31
4.21.   NADDD .....	32
4.22.   ND .....	32
4.23.   NE.....	32
4.24.   NS .....	33
4.25.   PC .....	33
4.26.   PNNNN.....	33
4.27.   R .....	33
4.28.   RESET .....	34

4.29.	RT .....	34
4.30.	SR .....	34
4.31.	SR0 .....	34
4.32.	SR1 .....	35
4.33.	RT8 .....	35
4.34.	RT16 .....	35
4.35.	S .....	35
4.36.	SCNNNNN .....	36
4.37.	SS .....	36
4.38.	SPHH:MM .....	37
4.39.	STHH:MM .....	37
4.40.	SV .....	37
4.41.	TEST .....	38
4.42.	TF .....	39
4.43.	TR .....	39
4.44.	TR0 .....	39
4.45.	WF .....	39
4.46.	X .....	40
<b>5.</b>	<b>MAINTENANCE.....</b>	<b>41</b>
5.1.	KEEPING THE INSIDE OF THE BOX DRY: .....	41
5.2.	CLEANING: .....	41
5.3.	BATTERIES: .....	41
<b>6.</b>	<b>TROUBLESHOOTING .....</b>	<b>43</b>
6.1.	UNIT WILL NOT RESPOND TO COMMUNICATIONS .....	43
6.2.	VIBRATING WIRE GAGE MEASUREMENT READS -999999.0 .....	43
6.3.	GAGE MEASUREMENT (ANALOG OR VIBRATING WIRE) READS -999999.9 .....	43
6.4.	VIBRATING WIRE GAGE READING IS UNSTABLE .....	43
6.5.	THERMISTOR MEASUREMENT SHOWS -99.9 DEGREES CELSIUS .....	43
	<b>APPENDIX A - SPECIFICATIONS.....</b>	<b>44</b>
A.1.	MEASUREMENT CAPABILITY .....	44
A.2.	POWER .....	44
A.3.	MEMORY .....	44
A.4.	CLOCK .....	44
A.5.	SERIAL INTERFACE (ALL LC-2 MODELS): .....	45
A.6.	RS-485 NETWORK .....	45
A.7.	VIBRATING WIRE MEASUREMENT .....	45
A.8.	INTERNAL/EXTERNAL TEMPERATURE MEASUREMENT .....	45
A.9.	MAIN BATTERY MEASUREMENT .....	45
	<b>APPENDIX B - CONNECTOR PINOUTS .....</b>	<b>46</b>
B.1	TRANSDUCER CONNECTIONS .....	46
B.1.1	<i>Transducer Cable Connections (8002-1-2).....</i>	<i>46</i>
B.1.2	<i>Transducer Cable Connections (8002-1-1).....</i>	<i>46</i>
B.2.	SENSOR CONNECTOR PIN-OUT (8002-1A-1, 8002-2A-1) .....	46
B.3.	COM CONNECTOR PIN-OUT .....	47
B.4.	RS-485 CONNECTOR PIN-OUT (OPTIONAL – 8002-1-3) .....	47
	<b>APPENDIX C - DATA FILE TRANSFER TO A WINDOWS PC .....</b>	<b>48</b>
C.1.	DOWNLOADING DATA USING LOGVIEW .....	48
C.2.	DOWNLOADING DATA USING HYPERTERMINAL (OR EQUIVALENT) .....	50
	<b>APPENDIX D - SAMPLE DATA FILES .....</b>	<b>52</b>
	<b>APPENDIX E - THERMISTOR TEMPERATURE DERIVATION.....</b>	<b>54</b>
	<b>APPENDIX F - NETWORKING .....</b>	<b>55</b>

F.1. DESCRIPTION .....	55
F.2. EXAMPLE OF A 4 DATALOGGER NETWORKING SESSION .....	56
<b>APPENDIX G – LOGWARE COMPATIBILITY .....</b>	<b>57</b>
G.1. BATTERY GAUGE: .....	57
<b>APPENDIX H – LITHIUM COIN CELL .....</b>	<b>58</b>
H.1. DESCRIPTION: .....	58
H.2. REPLACEMENT PROCEDURE: .....	58

## LIST of FIGURES, TABLES and EQUATIONS

TABLE 1 TRANSDUCER CABLE CONNECTIONS.....	3
EQUATION 1 - DISPLAYED GAGE READING USING LINEAR CONVERSION .....	25
EQUATION 2 - DISPLAYED GAGE READING USING POLYNOMIAL CONVERSION .....	26
TABLE 5 - LOGARITHMIC INTERVALS LIST .....	29
TABLE 6 - S COMMAND INFORMATION .....	36
TABLE 7 - SS COMMAND INFORMATION.....	36
TABLE 8 – TEST MENU INFORMATION .....	38
TABLE 9 - APPROXIMATE OPERATING TIMES .....	42
TABLE B-1A TRANSDUCER CABLE CONNECTIONS (USB DATALOGGER).....	46
TABLE B-1B TRANSDUCER CABLE CONNECTIONS (SERIAL DATALOGGER) .....	46
TABLE B-2 SENSOR CONNECTOR PIN-OUT .....	46
TABLE B-3 COM CONNECTOR PIN-OUT.....	47
TABLE B-4 RS-485 CONNECTOR PIN-OUT.....	47
EQUATION E-1 CONVERT THERMISTOR RESISTANCE TO TEMPERATURE .....	54
TABLE E-1 THERMISTOR RESISTANCE VERSUS TEMPERATURE .....	54
TABLE G-1 ACTUAL BATTERY LIFE VS. LOGWARE BATTERY GAUGE .....	57
<u>FIGURE 1 - TRANSDUCER CABLE CONNECTIONS .....</u>	<u>4</u>
<u>FIGURE 2 - SELECT WORKSPACE NAME .....</u>	<u>6</u>
<u>FIGURE 3 - SELECT WORKSPACE FOLDER.....</u>	<u>6</u>
<u>FIGURE 4 - LOGVIEW MAIN WINDOW .....</u>	<u>7</u>
<u>FIGURE 5 - LOGVIEW MAIN WINDOW WITH NEW PROJECT.....</u>	<u>8</u>
<u>FIGURE 6 - LOGVIEW CONTEXT MENU .....</u>	<u>9</u>
<u>FIGURE 7 - DATALOGGER SETTINGS, CONNECTION OPTIONS .....</u>	<u>9</u>
<u>FIGURE 8 - DATALOGGER HIGHLIGHTED, NOT CONNECTED.....</u>	<u>10</u>
<u>FIGURE 9 - DATALOGGER CONNECTION MISMATCH.....</u>	<u>11</u>
<u>FIGURE 10 - DATALOGGER CONNECTED.....</u>	<u>12</u>
<u>FIGURE 11 - DATALOGGER CONNECTION MISMATCH.....</u>	<u>12</u>
<u>FIGURE 12 - DEVICE MANAGER PORTS LIST.....</u>	<u>13</u>
<u>FIGURE 13 - HYPERTERMINAL CONNECTION DESCRIPTION.....</u>	<u>14</u>
<u>FIGURE 14 - COM PORT SELECTION .....</u>	<u>15</u>
<u>FIGURE 15 - COM PORT SETTINGS.....</u>	<u>15</u>
<u>FIGURE 16 - ORDER OF ARRAY USAGE.....</u>	<u>23</u>
<u>FIGURE 17 - LOGVIEW COLLECT DATA BUTTON.....</u>	<u>48</u>
<u>FIGURE 18 - DATA COLLECTION PROGRESS BAR.....</u>	<u>49</u>
<u>FIGURE 19 - DATA COLLECTION COMPLETE MESSAGE .....</u>	<u>49</u>
<u>FIGURE 20 - HYPERTERMINAL TRANSFER MENU .....</u>	<u>50</u>
<u>FIGURE 21 - SPECIFY DATA CAPTURE FILE .....</u>	<u>51</u>
<u>FIGURE 22 - HYPERTERMINAL/DATALOGGER COMMUNICATION .....</u>	<u>51</u>





## **1. INTRODUCTION**

The Model LC-2 Single Channel Datalogger is a low cost, battery powered and easy to use measurement instrument designed to read vibrating wire sensors equipped with thermistors.

The 320K standard memory provides storage for 16000 data. Each array consists of an optional datalogger ID string (16 characters maximum), a timestamp consisting of the year, date (julian day or month/day format), time (hhmm or hours/minutes format) and seconds when the reading was taken. Also included in the data is the internal 3V (or external 12V) battery voltage, the datalogger temperature, the vibrating wire reading, the temperature at the transducer and the array number in memory.

Internal math is calculated using 32 bit floating point notation (IEEE). Math operations on the instrument reading, such as application of a zero reading, gage factor (or calibration factor) and offset when using a linear conversion technique or polynomial coefficients when using the polynomial conversion, provide output directly in engineering units.

## **2. LC-2 MODEL TYPES**

### **2.1 RS-232 INTERFACE (8002-1-1, 8002-1A-1):**

The datalogger's internal configuration is defined through communication with a computer using the supplied RS-232 interface cable and LogView software. The datalogger may also be configured and monitored using any standard terminal emulator software, such as Microsoft Windows HyperTerminal™ or Geokon's Terminal Window Software. (See section 4, LC-2 Command List, if using terminal emulator software to interface to the datalogger.)

LC-2 model 8002-1-1 provides hard-wired transducer connection by means of an internal terminal strip (see Figure 1). LC-2 model 8002-1-1A provides transducer connection by means of a 10-pin connector. Refer to section 3.1, Transducer Installation to connect the vibrating wire transducer to the LC-2 datalogger.

### **2.2 USB INTERFACE (8002-1-2, 8002-1A-2):**

The datalogger's internal configuration is defined through communication with a computer using the supplied USB 2.0 interface cable and LogView software. The datalogger may also be configured and monitored using any standard terminal emulator software, such as Microsoft Windows HyperTerminal™ or Geokon's Terminal Window Software. (See section 4, LC-2 Command List, if using terminal emulator software to interface to the datalogger.)

When connected to a computer via the USB port, the LC-2 appears to the computer as a "virtual" COM port. The LC-2 datalogger also receives all of its operating power from the computer, thus extending the internal 3V (or external 12V) battery life. When disconnected from the USB port, the datalogger automatically switches to the internal 3V (or external 12V) battery pack.

LC-2 model 8002-1-2 provides hard-wired transducer connection by means of an internal terminal strip (see Figure 1). LC-2 model 8002-1A-2 provides transducer connection by means of a 10-pin connector. Refer to section 3.1, Transducer Installation to connect the vibrating wire transducer to the LC-2 datalogger.

### **2.3 RS-485 INTERFACE (8002-1-3, 8002-1A-3):**

The datalogger's internal configuration is defined through communication with a computer using the supplied RS-485 interface cable and LogView software. The datalogger may also be configured and monitored using any standard terminal emulator software, such as Microsoft Windows HyperTerminal™ or Geokon's Terminal Window Software. (See section 4, LC-2 Command List, if using terminal emulator software to interface to the datalogger.)

When connected to a computer via the 8001-5 (RS-232) or 8002-5(USB) RS-485 interface, up to 256 LC-2 dataloggers may be networked (daisy-chained) together over one RS-485 communications cable.

LC-2 model 8002-1-3 provides hard-wired transducer connection by means of an internal terminal strip (see Figure 1, pg. 4). LC-2 model 8002-1A-3 provides transducer connection by means of a 10-pin connector. Refer to section 3.1 Transducer Installation to connect the vibrating wire transducer to the LC-2 datalogger.

All data, both readings and configuration, are stored in non-volatile EEPROM with a typical storage life of 10 years (minimum). The internal temperature compensated real-time clock, used to provide timekeeping and triggering of readings, is accurate to  $\pm 2$  minutes/year.

The comma delineated ASCII output format allows for easy importing into popular spreadsheet programs such as Lotus 1-2-3™ or Microsoft Excel™. See Appendix D for sample data files.

For more information regarding setup and operation of the RS-485 LC-2 models, refer to APPENDIX F, NETWORKING.

### **3. GETTING STARTED**

The following equipment will arrive with the Model LC-2 datalogger;

1. Set of (2) alkaline 'D' cell batteries.
2. Set of (4) desiccant packs packaged with the batteries.
3. 9-pin Dsub to 10-pin Bendix RS-232 cable (included with models 8002-1-1, 8002-1A-1) or USB-A to 10-pin Bendix USB cable (included with models 8002-1-2, 8002-1A-2).
4. Model LC-2 Single Channel Datalogger Instruction Manual.

If any of these items are missing or damaged contact the factory for replacements. The following are optional accessories;

- RS-485 interface cable(s).
- S-8001-5 or S-8002-5 RS-485 computer interface.
- Vibrating Wire Sensor with built-in thermistor.

This section will outline the basic steps needed to install the communications software, establish communication with the Model LC-2 and configure the datalogger in the context of water level monitoring using a Geokon model 4500S Vibrating Wire Pressure Transducer.

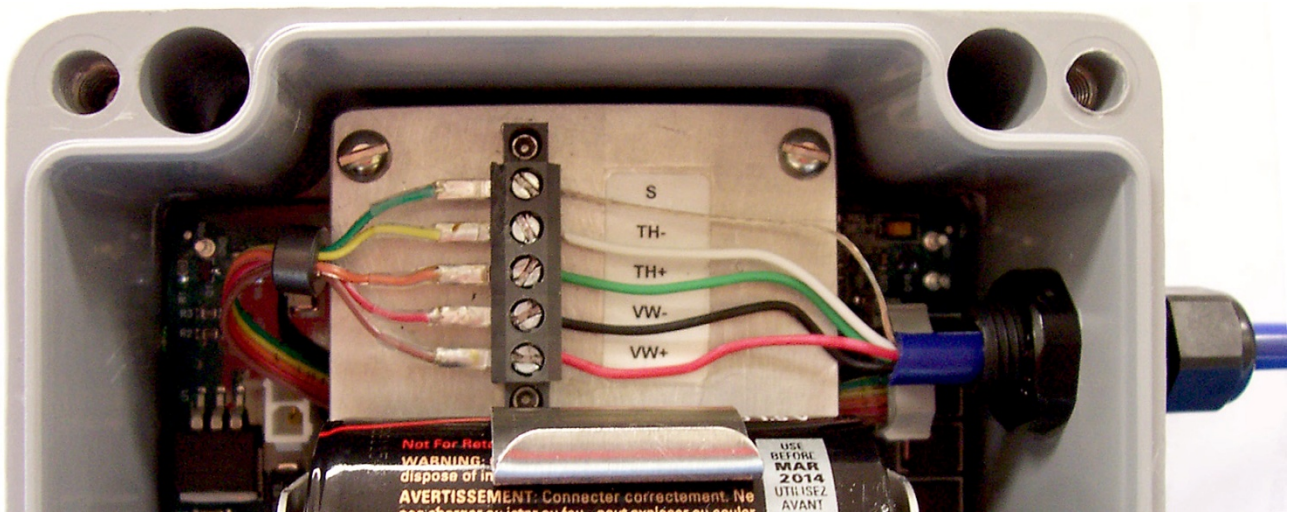
Open up the LC-2 by unscrewing the 4 captive screws on the top of the LC-2 enclosure. Make sure that no dirt, water or other contaminants are allowed to enter the LC-2 enclosure. Insert the 2 “D” cells straight down into the battery holder, while observing the correct polarity. Note that there is a ziplock bag containing 4 desiccant packs shipped along with the batteries. As soon as the batteries are installed, take the desiccant packs out of the ziplock bag and place them inside the enclosure. Immediately close and reseal the lid. This will help to prevent condensation of moisture within the enclosure.

#### **3.1. Transducer Installation:**

For LC-2 models 8002-1-1, 8002-1-2 and 8002-1-3, install the transducer to the LC-2 by removing the white plastic plug (which is used to keep the enclosure sealed during shipment), thread the transducers cable through the bulkhead fitting on the front of the LC-2 enclosure and wire the cables 5 conductors into the terminal strip per Table 1 and Figure 1:

<b>Terminal Strip Position</b>	<b>Cable Wire Color</b>	<b>Description</b>
VW+	RED	Vibrating Wire +
VW-	BLACK	Vibrating Wire -
TH+	GREEN	Thermistor +
TH-	WHITE	Thermistor -
S	BARE WIRE	Analog Ground (shields)

**Table 1 Transducer Cable Connections**



**Figure 1 - Transducer Cable Connections**

When complete, reinstall the top of the LC-2 enclosure.

Refer to APPENDIX B, CONNECTOR PINOUTS for LC-2s supplied with 10-pin Transducer Connector.

### 3.2. Software Installation and Setup

LogView is Graphical User Interface (GUI) software and is used to communicate with the datalogger, using a personal computer running a Microsoft Windows® operating system. Other general purpose communication programs (i.e. Windows HyperTerminal™) can also be used to communicate with the Model LC-2 via text-based commands. The LogView and USB drivers install program can be downloaded at [www.geokon.com/software](http://www.geokon.com/software).

Perform the following steps to install LogView software for each computer that will connect to an LC-2. These instructions are for computers running Windows XP. The installation procedure is very similar for computers running Windows7, Windows 2000 and Windows 98. This installation procedure needs to be performed just once for each computer that will run LogView to communicate with a LC-2 datalogger.

**NOTE:** The USB drivers are only required for LC-2 models 8002-1-2, 8002-1A-2 and the 8002-5 RS-485 Interface

Make sure that the (2) 1.5V D-cell alkaline batteries are installed in the datalogger (See section 5.3 BATTERIES for instructions) and that the LC-2 datalogger is not connected to the computer at this time:

#### **3.2.1. LogView Installation:**

1. Using Windows Explorer, navigate to the extracted downloaded files. Double click on the file “start.bat” to start the install process.
2. Click “**Next >**” when the **Welcome** window appears.
3. When the **Choose Install Location** window appears, choose a folder for the LogView installation then click “**Next>**”.
4. When the **Choose Start Menu Folder** window appears, choose an appropriate folder (default is Geokon) then click “**Install**”.
5. Click “**Next >**” when the **Java Installation Complete** window appears.
6. Click “**Finish**” when the **Completing the LogView Setup Wizard** window appears.
7. Remove the LogView installation CD from the computers CD drive.

#### **3.2.2. Launching LogView:**

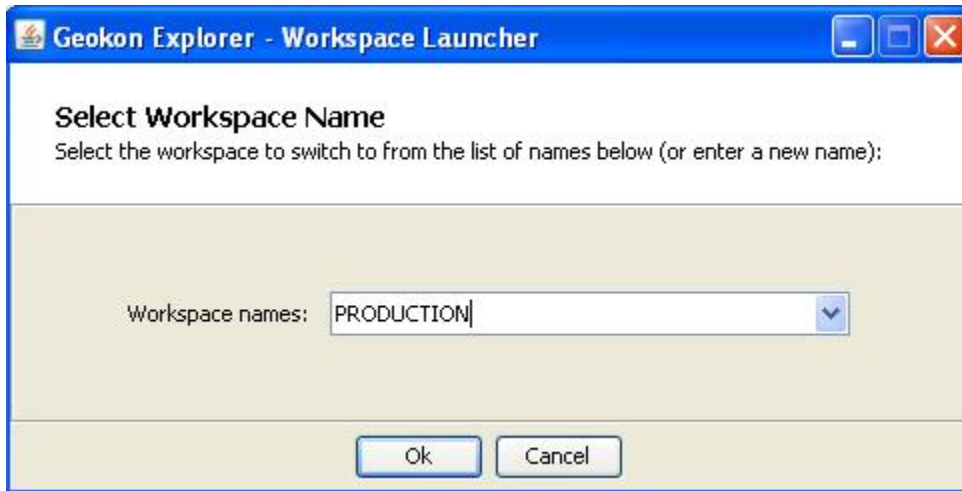
Launching LogView can be accomplished two different ways. Double clicking on the desktop icon:



Or via the Start button: “Programs → Geokon → LogView”

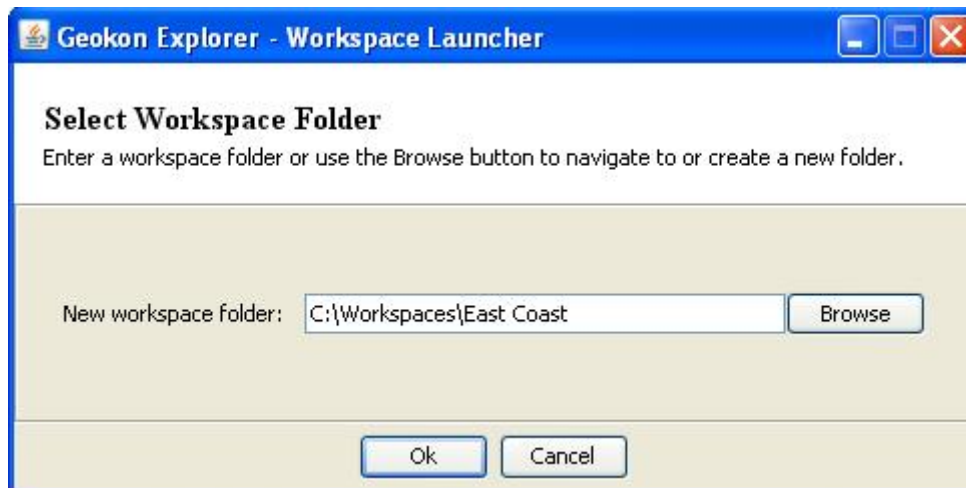
### **3.2.3 LogView Workspaces:**

When opening LogView for the first time, the user will be prompted to create a workspace name (see figure 2). The workspace name can be any combination of letters and numbers and, ideally, will be descriptive in nature. See the LogView User's Guide for more information on workspaces.



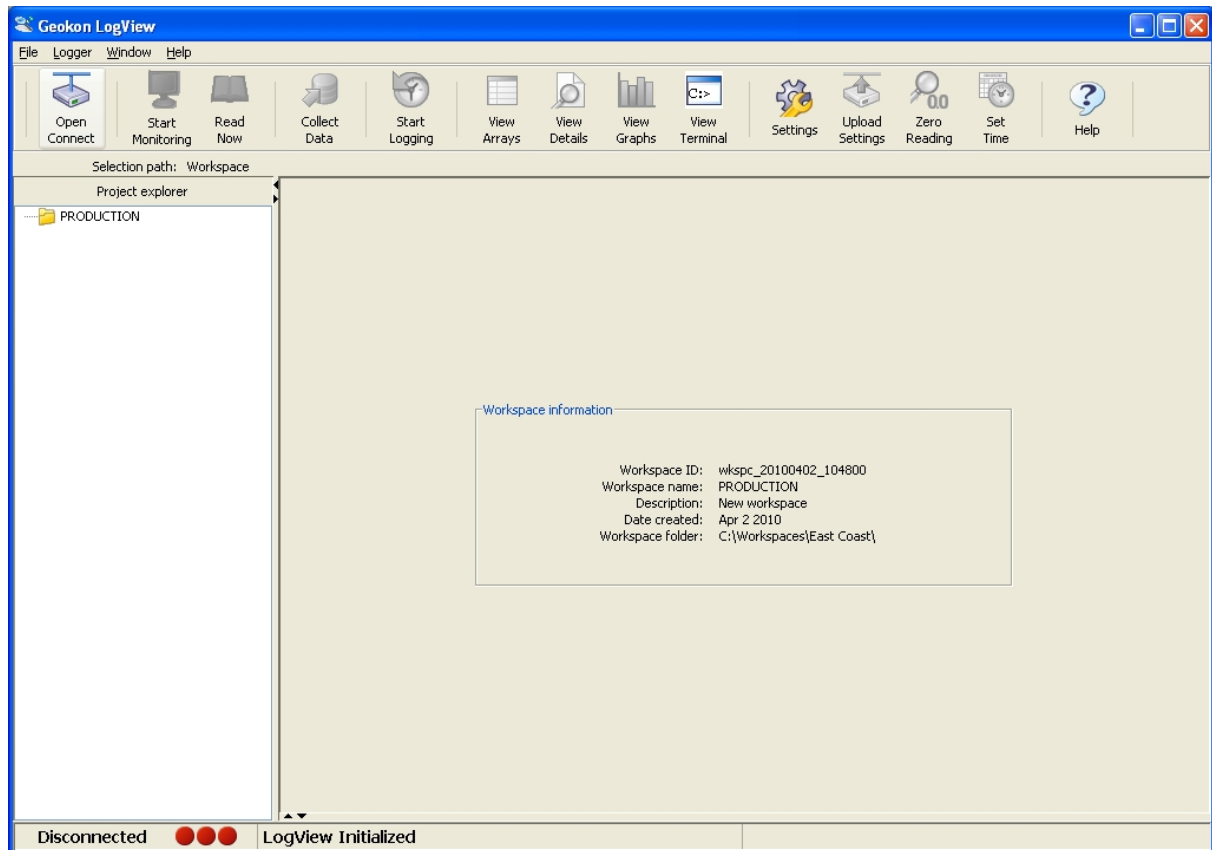
**Figure 2 - Select Workspace Name**

Once the workspace name has been selected, clicking on “**Ok**” causes LogView to prompt the user to choose or create a folder where all the workspace elements will be stored (see figure 3). The folder location may be entered directly, i.e., C:\Workspaces\East Coast or the **Browse** button may be used to navigate to a folder location or to create a new folder (see below). This workspace location will be stored in the LogView configuration for subsequent application access. Once workspaces are created, future user access is always by name.



**Figure 3 - Select Workspace Folder**

If no other is specified, a default folder path is displayed based on the system default workspace path combined with the new workspace name. After the folder path has been specified, either the default or user selected (see figure 3), clicking on “**Ok**” will display the main window of LogView (see figure 4). On the left-hand side of the main window is the Project Explorer displaying the newly created workspace. The user can now add new project(s), datalogger(s) and sensor configurations to the workspace by right-clicking on the workspace and using the menu tools.

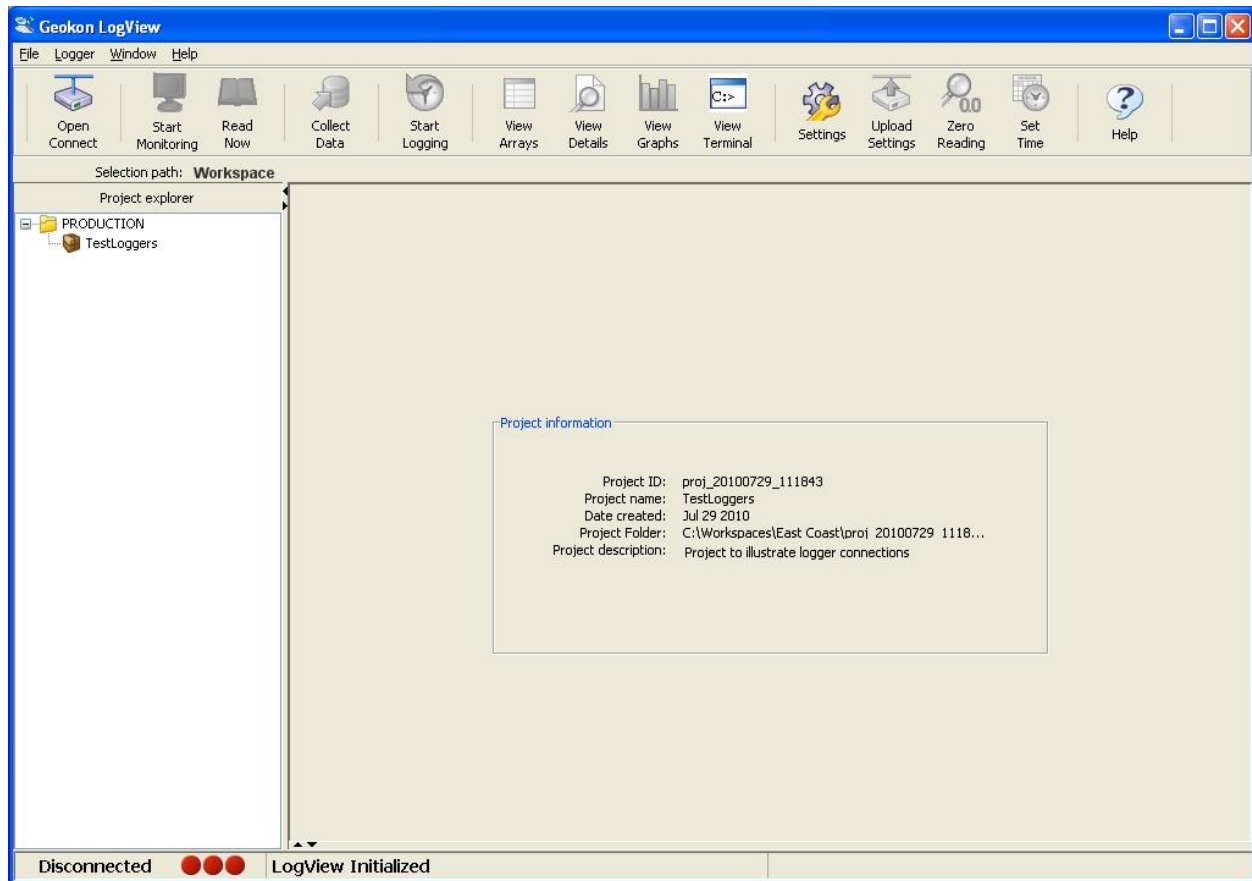


**Figure 4 - LogView Main Window**

### **3.2.4 Adding Projects to LogView Workspaces:**

Right-clicking on the “**PRODUCTION**” workspace brings up a context sensitive menu that allows the user to add projects to this workspace (using the “**New->Project**” menu selection). Select a name that makes sense for the real-world project this program will be used for. In this example “TestLoggers” was chosen as the project name (see figure 5 below).



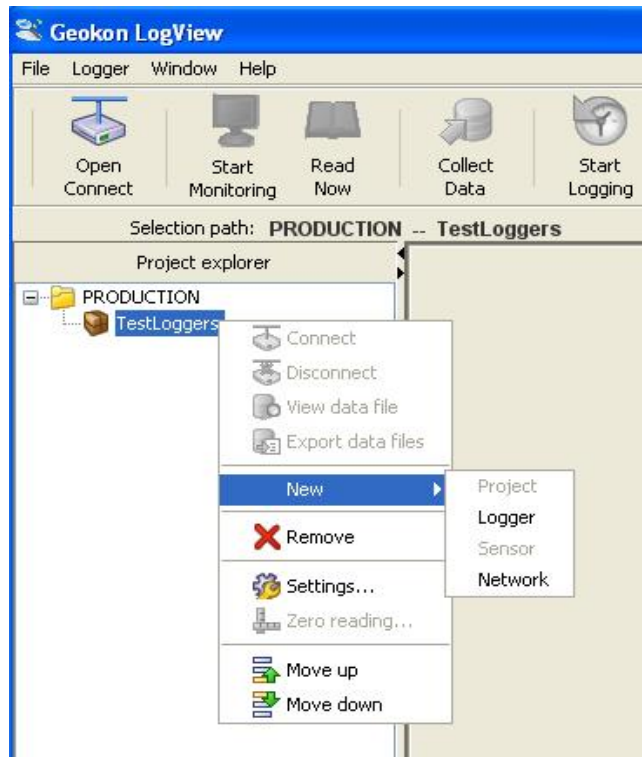


**Figure 5 - LogView Main Window with new project**

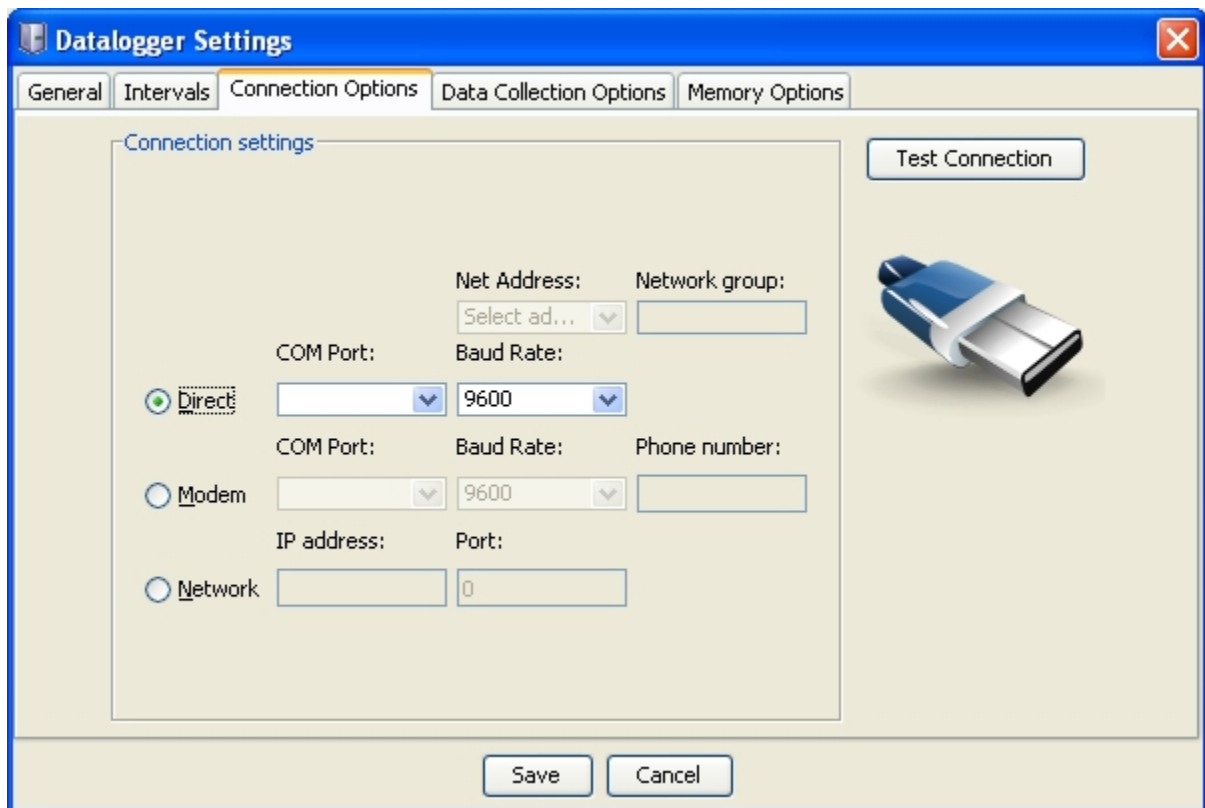
### **3.2.5 Adding Dataloggers to LogView Projects:**

Right-clicking on the “**TestLoggers**” project brings up a context sensitive menu (see figure 6) that allows users to add dataloggers to their projects. Selecting **New->Logger** from the context menu causes the “**Datalogger Settings**” dialog to be displayed. Like Workspaces and Projects, Dataloggers can be assigned a unique human-readable name. For this example, “**MyLogger**” was chosen for the Datalogger name. For a complete description of all datalogger settings please see the LogView Online Help section on Datalogger Settings. For connection purposes, the relevant tab in this dialog is “**Connection Options**” (see figure 7).

Once connected to a PC, all LC-2 dataloggers require a COM port to be identified in the “**Connection Options**”. Starting with firmware revision 5.2.X, LC-2 dataloggers can communicate at baud rates of 9600 and 115,200. Before this revision the datalogger baud rate was 9600 only so, for these dataloggers, the default setting should not be changed (See figure 7).



**Figure 6 - LogView Context Menu**



**Figure 7 - Datalogger Settings, Connection Options**

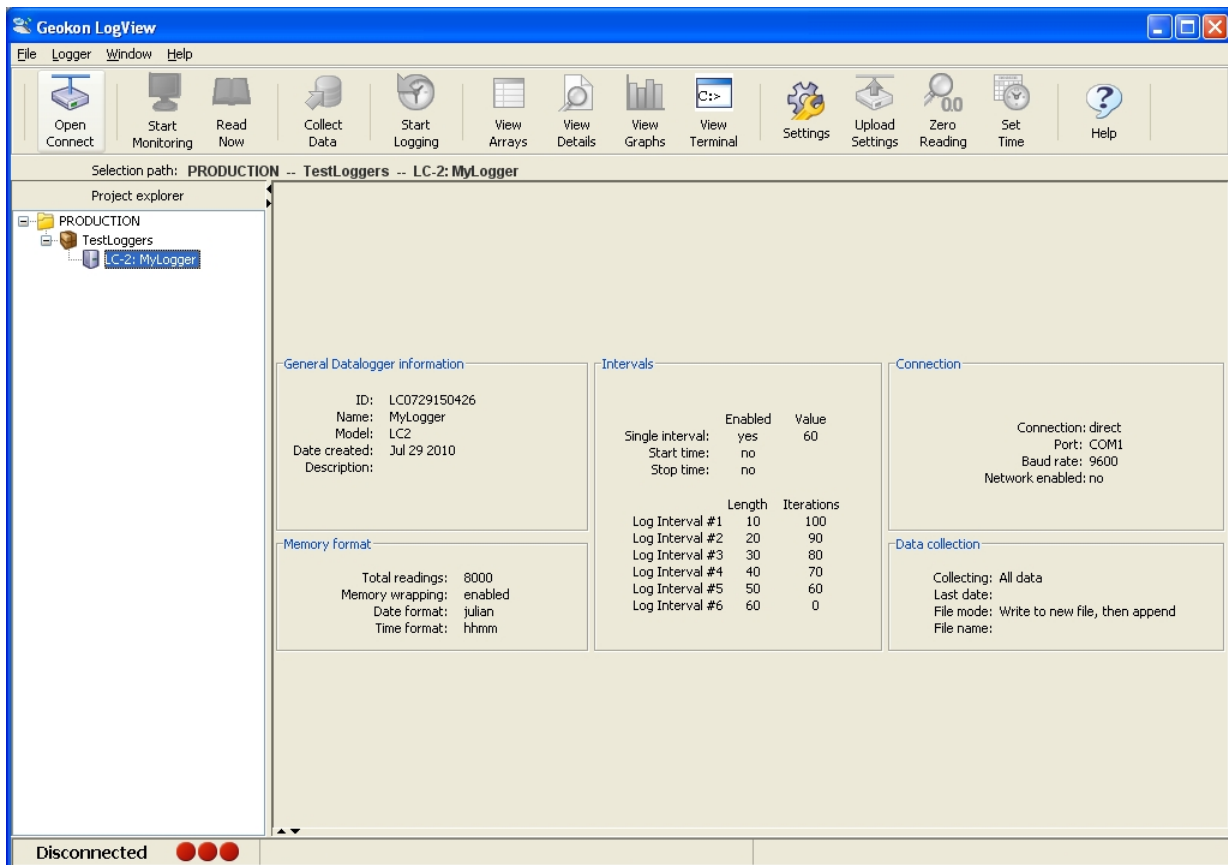
### **3.2.6. LC-2 Connection (8002-1-1, 8002-1A-1):**

Connect the supplied LC-2 RS-232 Communications cable (COM-108) to the COM port of the LC-2 datalogger. The protective cap on the datalogger COM connector is removed by pushing in and turning. Plug the DB-9 end of the RS-232 Communications cable into the host computer's RS-232 port (either internal or external via a USB to Serial converter). Proceed to section 3.2.8, Connecting to a Datalogger with LogView

### **3.2.7. LC-2 Connection (8002-1-2, 8002-1A-2):**

Connect the supplied LC-2 USB Communications cable (COM-109) to the USB port of the LC-2 datalogger. The protective cap on the datalogger USB connector is removed by pushing in and turning. Plug the USB-A end of the USB cable into an available USB-2.0 port on the host computer.

**NOTE:** On certain PCs with operating systems older than XP, Service Pack 3, the 8002-1-2 may require the installation of a driver to properly communicate with the PC. If the PC does not recognize the datalogger's internal USB to serial converter then the driver may be installed by executing the program CDMxxxxx.exe on the LogView Install CD.



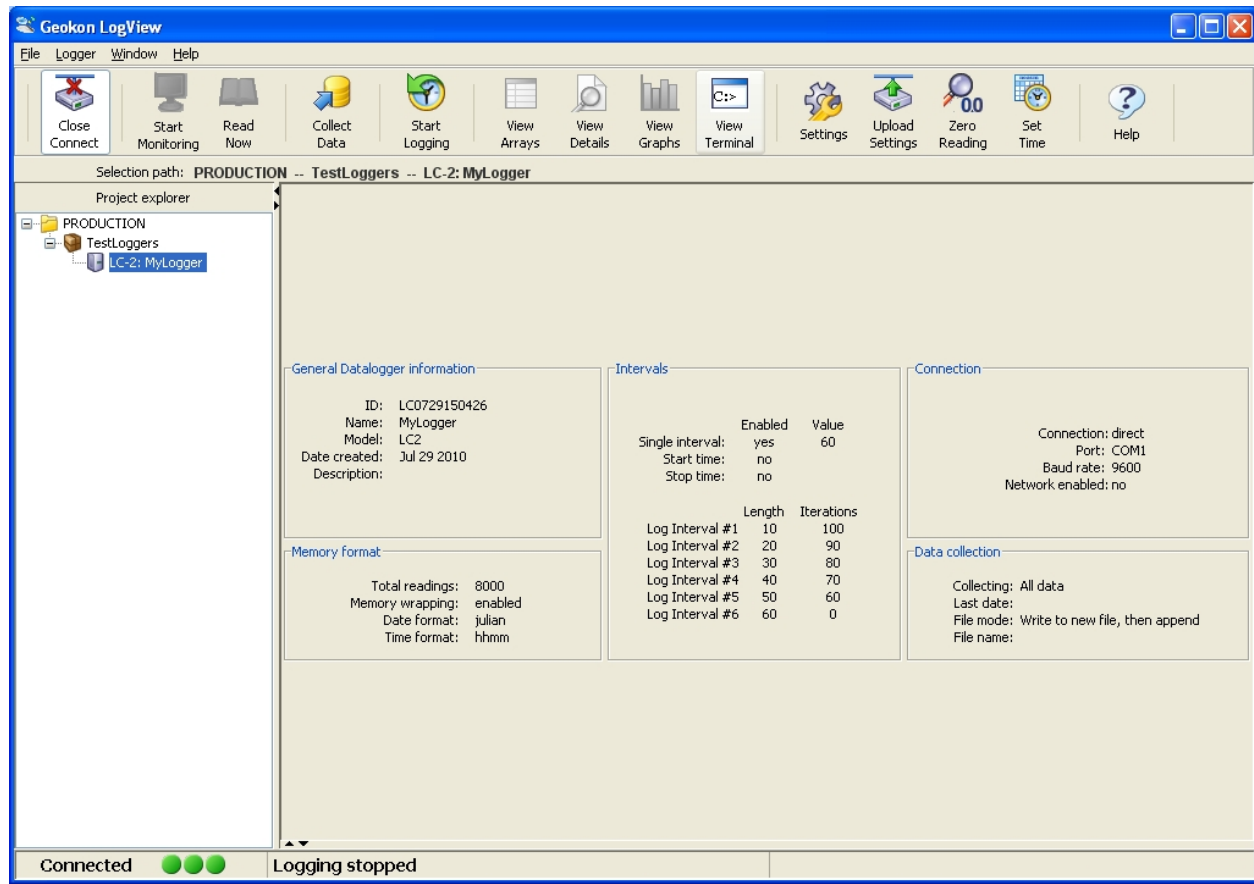
**Figure 8 - Datalogger Highlighted, Not Connected**

### **3.2.8. Connecting to a Datalogger with LogView:**

1. With a Datalogger profile configured and selected in the Project Explorer (see figure 8), click on the “**Open Connect**” button on the LogView Toolbar.
2. When connecting to a new datalogger for the first time, the message below (see figure 9) may be displayed after a few seconds. This is normal and is only an indication that the datalogger doesn’t match the configuration created in the Project Explorer. Click on “Continue” to finish connecting to the datalogger.
3. Click on the “**Upload Settings**” button on the LogView Toolbar to synchronize the datalogger with the LogView configuration (see figure 10).
4. LogView is now connected and configured correctly for the LC-2 datalogger. Sensors can now be added to the datalogger in a similar fashion as adding Dataloggers to Projects. Sensor settings are accessed via the context menu from the Project Explorer.



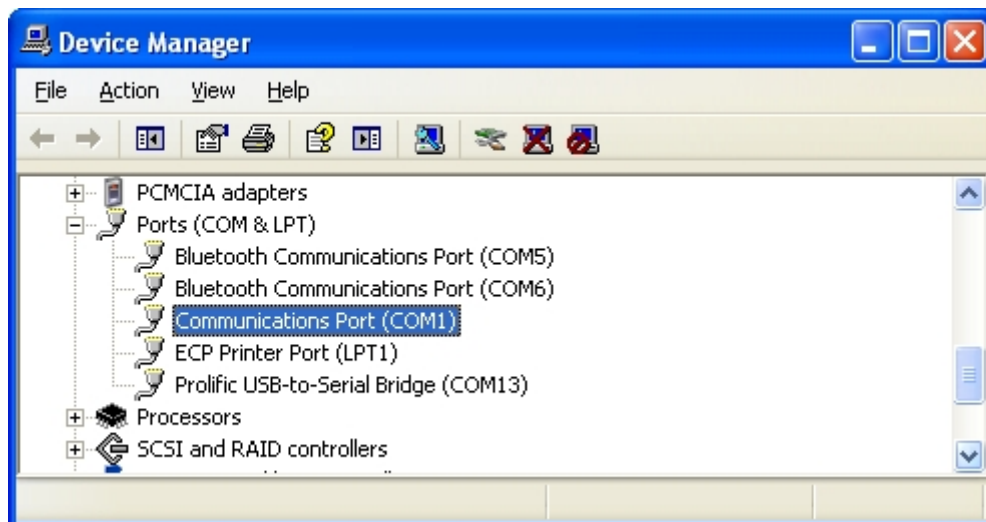
**Figure 9 - Datalogger Connection Mismatch**



**Figure 10 - Datalogger Connected**

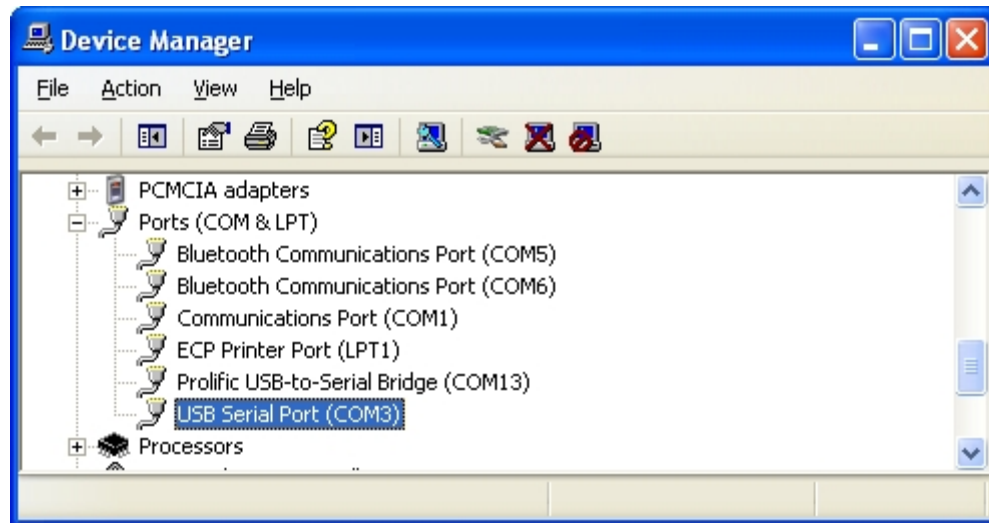
### **3.2.9. Determining COM Port Numbers:**

When connecting an 8002-1-1 or 8002-1A-1 datalogger to a PC with an internal serial port(s) the COM Port number that LogView requires is usually COM1 or COM2 but, occasionally may be COM3 if the PC has more than one internal serial port. The figure below (see figure 11) illustrates that the PC has 2 serial ports, one internal, COM1 and the other via a USB to serial converter, COM13



**Figure 11 - Datalogger Connection Mismatch**

When connecting an 8002-1-2 or 8002-1A-2 datalogger to a PC then the COM Port number that LogView requires can be any number and depends on how many other devices are attached to the PC such as, internal serial ports and Bluetooth devices. The figure below (see figure 12) illustrates that the PC has 3 serial ports, one internal, COM1 and the other two via USB to serial converters, COM13 and COM3. One way to determine which COM port an 8002-1-2 datalogger is attached to is to disconnect the cable and see which COM device disappears from the Device Manager Ports list.



**Figure 12 - Device Manager Ports List**

In this case, COM3 is actually the 8002-1-2 datalogger and not a universally available serial port.

### 3.3. Example Setup Using Text Comands

**For 8002-1-1 and 8002-1-2:** Route the vibrating wire transducer cable into the LC-2 enclosure through the SENSOR bulkhead fitting. Referring to Table 1, Figure 1 or Appendix B.1A, Transducer Cable Connections, connect the cable wires to the datalogger's 5 pin internal terminal strip, located on the battery pack mounting plate.

**For 8002-1A-1 and 8002-1A-2:** Connect the vibrating wire transducer cable to the SENSOR connector on the LC-2 enclosure.

For USB connected LC-2 dataloggers (8002-1-2, 8002-1A-2), it is important that the LC-2 first be connected to the computers USB port before running LogView (or other communications program) so that the LC-2 can be recognized by the computer as a virtual COM port.

Proceed with the following steps to connect with the datalogger using a terminal emulator program such as Microsoft Windows HyperTerminal™:

1. Launch HyperTerminal (Start → All Programs → Accessories → Communications → HyperTerminal).  
If running under Vista or newer, contact Geokon for Geokon's Terminal Window Software.
2. Enter a name for the New Connection and click OK (see Figure 13):



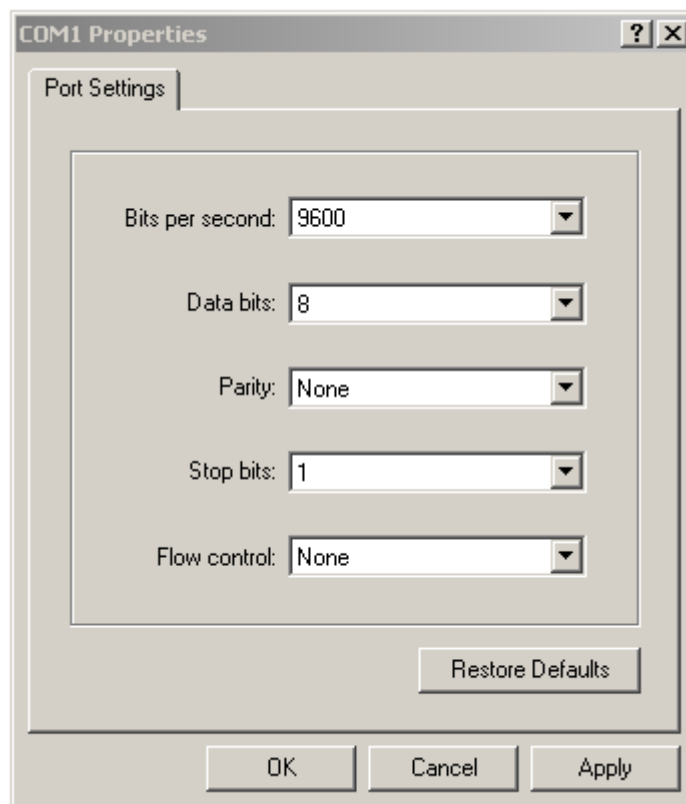
**Figure 13 - HyperTerminal Connection Description**

3. In the Connect Using window, select the appropriate COM port:



**Figure 14 - COM Port Selection**

4. In the COM Properties window, configure the COM port:



**Figure 15 - COM Port Settings**



8002-4-1 (RS-232): Configure the COM port (typically COM1 or COM2) as 9600 Bits per second, 8 Data bits, no Parity, 1 Stop bit, no Flow control.

8002-4-2 (USB): Configure the new COM port that is added when the LC-2 is connected as 9600 Bits per second, 8 Data bits, no Parity, 1 Stop bit, no Flow control.

5. Click Apply then OK.
6. Press <ENTER> to wake the datalogger from sleep. The datalogger returns the power up prompt;

**Hello. Press "?" for Help.**  
\*

**Note: If no characters are received in 15 seconds the datalogger (non-networked) will return to its low power sleep mode. Press <ENTER> to wake it again.**

**Note: When network commands are enabled and RS-485 is being used, the address must be sent before the respective datalogger will respond. See sections 4.20 - 4.24 for additional information.**

7. Type ? <ENTER> to display the Help list. See section 4 for detailed information on all the commands listed. **All commands must be entered in capital letters!**

\*?

Command	Description
-----	-----
C	view current Clock
CSmm/dd/yy/hh:mm:ss	Clock Set
DEFAULT	Load factory DEFAULT settings
DF	Date Format(0=julian,1=month,day)
Dnnnnn	Display nnnnn arrays from pointer
E	End communications and go to sleep
Gnn/szzzz/sffff/soooo	Gage information, where; nn = gage type szzzz = zero reading with sign sffff = gage factor with sign soooo = offset with sign
IDdddddddddddddd	view current ID,set to ddddddddddddddd
LC	select Linear Conversion
Ln/llll/iii	view Log intervals/change n interval llll = length iii = iterations of interval
LD,LE	Log intervals Disable, Enable
M,MD,ME	Monitor status, Disable, Enable
MS	Memory Status
N	Display Next time to read
NAddd	Network Address (1-256)
NS,ND,NE	Network Status, Disable, Enable
PC	select Polynomial Conversion
Pnnnnn	Position array pointer to nnnnn

<b>R</b>	<b>Reset memory</b>
<b>RESET</b>	<b>RESET processor</b>
<b>RT</b>	<b>Readings Total (8=8000 readings,16=16000 readings)</b>
<b>SR</b>	<b>Synchronize Readings (0=not synch'd,1=synch'd)</b>
<b>S,SS</b>	<b>datalogger Status, System Status</b>
<b>SCnnnnn</b>	<b>view SCan interval/enter nnnnn interval</b>
<b>SPhh:mm</b>	<b>StoP logging, hh:mm = stop time</b>
<b>SThh:mm</b>	<b>STart logging, hh:mm = start time</b>
<b>SV</b>	<b>Software Version</b>
<b>TEST</b>	<b>System Test</b>
<b>TF</b>	<b>Time Format(0=hhmm,1=hh,mm)</b>
<b>TR,TR0</b>	<b>display TRap count, zero TRap count</b>
<b>WF</b>	<b>Wrap Format(0=don't wrap memory, 1=wrap memory)</b>
<b>X</b>	<b>Single Reading - NOT stored</b>
<b>*</b>	

8. Type R and <ENTER> to reset the memory pointers. Type Y <ENTER> to the confirming "Are you sure(Y/N)?" question. The datalogger should respond with "Memory Cleared";

```
*R
Are you sure(Y/N)?Y
Memory cleared.
*
```

9. Type C <ENTER> to display the current real time clock setting. See section 4.3. if adjustments need to be made;

```
*C
Date: 04/03/07 Time: 15:51:50
*
```

10. Next, the configuration for the type of gage being read must be specified. See section 4.8 for additional information.

Assume a Geokon Model 4500S-50 Vibrating Wire Pressure Transducer with a gage factor of 0.01234 psi/digit (found on the calibration sheet). Multiply this value by 2.31 to convert psi to feet of water, resulting in a factor of 0.02851. As with all vibrating wire gage measurements, a zero reading needs to be determined for proper operation. Enter the gage type and clear the zero reading, gage factor and offset positions in the datalogger's memory using the G command;

```
*G1/0/1/0
GT: 1 ZR: 0.00000 GF: 1.00000 GO: 0.00000
*
```

11. Make sure logarithmic intervals are disabled by typing LD and <ENTER>;

```
*LD
Log intervals disabled.
*
```

12. Enter a scan interval of 10 seconds by typing **SC10** and <ENTER>;

```
*SC10
Scan interval: 10 second(s).
*
```

13. Enable real-time display of the readings by typing **ME** <ENTER>;

```
*ME
Monitor mode enabled.
*
```

14. Start datalogging with the **ST** command;

```
*ST
Logging started.
*
```

Every 10 seconds an array of readings will display. For example;

```
2007,348,1217,32,3.11,23.50,-9149.485,21.6,1
*2007,348,1217,40,3.11,23.59,-9149.782,21.6,2
*2007,348,1217,50,3.11,23.67,-9149.659,21.6,3
2007,348,1218,0,3.11,23.41,-9149.812,21.6,4
2007,348,1218,10,3.11,23.19,-9149.694,21.6,5
```

Each line displayed represents an array of data, one set of readings taken at that interval. The seventh value in each line represents the gage reading of the transducer.

See APPENDIX D – SAMPLE DATA FILES for more information on the array format.

15. The transducer must be positioned to determine a zero reading. Follow the instructions in the Piezometer Instruction Manual for saturating the filter and lowering into the well. Let the transducer come to thermal equilibrium by leaving it immersed in the water for 15-20 minutes. If absolute depth of water is desired, position the transducer just above the water level. Note the displayed reading in the array. If change (delta) in water level is desired, note the reading displayed with the transducer left at its immersed location.

For the purpose of this example, assume that the transducer will monitor absolute depth, has been pulled out of the water, and the reading displayed is **-9896.820**. Ignoring the sign and digits to the right of the decimal point yields an offset of 9896 to be entered into the datalogger's memory. Press **SP** <ENTER> to stop logging. Enter the zero reading and gage factor by typing **G/9896/0.02851** <ENTER>. Lower the transducer back into the well to its installed location (below the maximum expected drawdown).

```
*G/9896/0.02851
GT: 1    ZR: 9896.00    GF: 0.02851    GO: 0.00000
*
```

16. Set the scan interval with the **SC** command. Scan interval is in seconds (3-86400).

```
*SC3600
Scan interval: 3600 second(s).
*
```

17. Press **ST**<ENTER> to start logging;

```
*ST
Logging started.
2009,93,1621,55,3.22,25.30,-0.582,25.0,1
```

The datalogger will continue to log values based on the entered scan interval until one of the following conditions is met:

1. The battery goes dead.
2. The stop command is issued (section 4.38).

18. Press **E** <ENTER> to end communications with the datalogger and enter the low power sleep mode.

*Consult the LogView manual or see APPENDIX C, DATA FILE TRANSFER TO WINDOWS PC of this manual in regard to collecting data.*

#### **4. COMMAND LIST**

The commands listed here are to be used if communications between the LC-2 and the host computer are established via a terminal emulator (i.e. Windows HyperTerminal). If using LogView, these commands can be ignored. To send commands and receive information from the Model LC-2 the communications mode must be established between the host computer and the datalogger (see section 3.2 and section 3.3).

Pressing ? <ENTER> while in the communications mode displays this list of commands:

<b>Command</b>	<b>Description</b>
<b>C</b>	<b>view current Clock</b>
<b>CSmm/dd/yy/hh:mm:ss</b>	<b>Clock Set</b>
<b>DEFAULT</b>	<b>Load factory DEFAULT settings</b>
<b>DF</b>	<b>Date Format(0=julian,1=month,day)</b>
<b>Dnnnnn</b>	<b>Display nnnnn arrays from pointer</b>
<b>E</b>	<b>End communications and go to sleep</b>
<b>Gnn/szzzz/sffff/soooo</b>	<b>Gage information, where;</b> nn = gage type szzzz = zero reading with sign sffff = gage factor with sign soooo = offset with sign
<b>IDdddddddddddddd</b>	<b>view current ID,set to ddddddddddddddd</b>
<b>LC</b>	<b>select Linear Conversion</b>
<b>Ln/llll/iii</b>	<b>view Log intervals/change n interval</b> llll = length iii = iterations of interval
<b>LD,LE</b>	<b>Log intervals Disable, Enable</b>
<b>M,MD,ME</b>	<b>Monitor status, Disable, Enable</b>
<b>MS</b>	<b>Memory Status</b>
<b>N</b>	<b>Display Next time to read</b>
<b>NAddd</b>	<b>Network Address (1-256)</b>
<b>NS,ND,NE</b>	<b>Network Status, Disable, Enable</b>
<b>PC</b>	<b>select Polynomial Conversion</b>
<b>Pnnnnn</b>	<b>Position array pointer to nnnnn</b>
<b>R</b>	<b>Reset memory</b>
<b>RESET</b>	<b>RESET processor</b>
<b>RT</b>	<b>Readings Total(8=8000 readings,16=16000 readings)</b>
<b>SR</b>	<b>Synchronize Readings (0=not synch'd,1=synch'd)</b>
<b>S,SS</b>	<b>datalogger Status, System Status</b>
<b>SCnnnnn</b>	<b>view SCan interval/enter nnnnn interval</b>
<b>SPhh:mm</b>	<b>StoP logging, hh:mm = stop time</b>
<b>SThh:mm</b>	<b>STart logging, hh:mm = start time</b>
<b>SV</b>	<b>Software Version</b>

<b>TEST</b>	<b>System Test</b>
<b>TF</b>	<b>Time Format(0=hhmm,1=hh,mm)</b>
<b>TR,TR0</b>	<b>display TRap count, zero TRap count</b>
<b>WF</b>	<b>Wrap Format(0=don't wrap memory,1=wrap memory)</b>
<b>X</b>	<b>Single Reading - NOT stored</b>

All of these commands are executed by typing with the correct syntax and pressing <ENTER>. If the command has not been entered correctly, the datalogger will usually not respond. For example;

```
*L7/100/255
*
```

The datalogger will respond to correctly entered commands by displaying the modified values. The purpose and syntax of each of these commands are discussed in the following sections.

#### 4.1. BRnnn

This command is not displayed in the help text (?<ENTER>). Displays or sets the current baud rate. Valid numbers for “nnn” are 9 (9600 baud) or 115 (115,200 baud). The example below changes the baud rate to 9600 with no confirmation.

```
*BR9
```

#### 4.2. C

Display the current datalogger real-time clock settings. The **CS** command section explains how to adjust the clock settings.

```
*C
Date: 02/21/07   Time: 10:43:08
*
```

#### 4.3. CSmm/dd/yy/hh:mm:ss

Set the datalogger’s internal real time clock; mm represents the month, dd the day of the month, yy the year, hh the hours, mm the minutes, and ss the seconds. Leading zeros are not needed except on the minutes and seconds entries. Illegal combinations will be ignored (i.e. CS02/30/97 or CS///12:60). Fields can be left blank to avoid changing (i.e. CS//97 to just change the year).

```
*CS//10:45:00
Date: 02/21/07   Time: 10:45:00
*
```

Note:

If logging is currently started and the clock is changed, a restart of the scan interval or log interval table will occur.

If the scan interval is fast (i.e. 3 seconds), logging may need to be stopped and re-started, depending on whether or not the internal “next time to read” register becomes unsynchronized with the current time. This is true with both the command line interface and setting the datalogger clock within LogView.

#### 4.4. DEFAULT

Load the factory default settings. All stored readings as well as the ID, networking and real-time clock settings are not affected by this command. User will be asked to verify before executing. Press **Y** to continue, any other key to abort.

```
*DEFAULT
This will load the factory default settings!
Are you sure(Y/N)?Y
Restored to factory default settings.
*
```

LC-2 setup after DEFAULT has been issued:

```
*S
MS:0  OP:1  UP:1
GT: 1  ZR: 0.00000  GF: 1.00000  GO: 0.00000
Scan interval: 3 second(s).
Logging stopped.
Log intervals disabled.
Monitor mode enabled.
      *SS
Signature of RAM1: 58294
Signature of RAM2: 47463
Signature of RAM3: 51545
Signature of RAM4: 15546
Signature of ROM: 4497
Trap count: 0
Network address: 1
Network recognition disabled.
Time format is hhmm.
Date format is julian.
*
*WF
Logging will not stop when memory is full
*
*SR
Readings are synchronized to the top of the hour.
*
*RT
8000 readings maximum
*
*L
      Log Intervals List
-----

Interval #1 Length: 10    Iterations: 100

Interval #2 Length: 20    Iterations: 90

Interval #3 Length: 30    Iterations: 80

Interval #4 Length: 40    Iterations: 70

Interval #5 Length: 50    Iterations: 60

Interval #6 Length: 60    Iterations: 0

*
```

## 4.5. DF

Display or set the date format. This setting determines how the date information will be displayed in the array when the monitor mode is active or arrays are displayed from memory. Entering DF displays the current date format. Entering DF0 sets the date format to julian. Entering DF1 sets the date format to month,day. The default date format display is Julian (decimal) day.

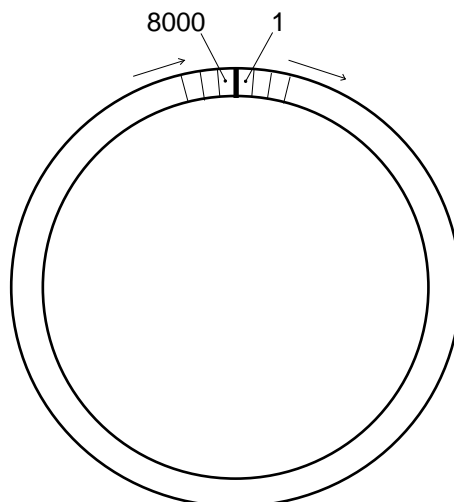
```
*DF
Date format is julian.
*X
2009,52,1343,20,3.00,24.7,-5372.293,24.3
*DF1
Date format is month,day.
*X
2009,2,21,1343,25,3.00,24.7,-5372.293,24.3
*DF0
Date format is julian.
*X
2009,52,1343,30,3.00,24.7,-5372.293,24.3
```

## 4.6. Dnnnn

Use the D command to display arrays forward from the User Position for verification or collection. The updated memory pointers are displayed by this command.

```
*D
MS:8000   OP:1567   UP:1006
*
```

MS represents the Memory Status of the datalogger. This number indicates how many arrays have been written to memory. If, as in the above example, it is at 8000 and WF (wrap format) = 1, then memory has been filled and it is now over-writing the oldest arrays. If it is at 8000 and WF = 0, then the memory has been filled and logging has stopped. Figure 3 illustrates the ring memory scheme.



**Figure 16 - Order of Array Usage**



OP represents the Output Position that the next array will be written to.

UP represents the User Position. This value is updated by D and P commands. The user may display arrays from this position or re-position to another array. The last column in the data array is the User Position.

Display nnnn arrays from the current User Position. Press any key (other than a letter or digit) to abort the display

```
*P3600
MS:8000 OP:3683 UP:3600
*D5
2009,94,0838,28,3.22,22.23,9978.889,25.0,3600
2009,94,0838,33,3.22,22.23,9978.917,25.0,3601
2009,94,0838,38,3.22,22.23,9979.131,25.0,3602
2009,94,0838,43,3.22,22.23,9979.185,25.0,3603
2009,94,0838,48,3.22,22.23,9979.192,25.0,3604

MS:8000 OP:3683 UP:3605
*
```

The format is comma delineated ASCII, identical to that displayed when the Monitor mode is active. See Appendix D for a sample data file. See Appendix C in regards using the D command to collect data. When the array display is finished the memory pointers are displayed.

## 4.7. E

Returns the datalogger to its low power sleep mode (readings continue to be logged and displayed in this mode).

**This command should always be used when finished communicating with the datalogger to ensure the lowest power consumption.** However, the datalogger (non-networked) will enter sleep mode regardless if no command is received in a period of approximately 15 seconds, or immediately after the 2<sup>nd</sup> reading (whichever comes first).

To return from the low power operating mode press <ENTER>. The datalogger responds;

```
Hello. Press "?" for Help.
*
```

#### 4.8. Gnn/szzzz/smmmm/soooo or Gnn/saaaa/sbbbb/scccc

When using linear conversion (LC) of the instrument reading, the G command is used to select the gage type and enter the gage zero reading, gage factor, and gage offset. When using polynomial conversion (PC), the G command is used to select the gage type and enter the three polynomial coefficients, A, B and C. Entering just G will return the current gage information. For example, with linear conversion selected;

```
*G
GT: 1   ZR: 0.00000   GF: 1.00000   GO: 0.00000
*
```

With polynomial conversion selected;

```
*G
GT: 1   PA: 0.00000   PB: 1.00000   PC: 0.00000
*
```

Note the change in response depending on the conversion method selected.

Slashes (/) are entered to delineate the values and to substitute for a value that will not be changed. For example;

```
*G
GT: 1   ZR: 0.00000   GF: 1.00000   GO: 0.00000
*G//1234.5
GT: 1   ZR: 0.00000   GF: 1.00000   GO: 1234.500
*
```

##### Linear Conversion

The command is described further as follows; **nn** represents the gage type, or the configuration of the datalogger's input channel (see Tables 1&2 in this section), **szzzz** represents the zero reading for the transducer being read, **smmmm** represents the multiplier (calibration or gage factor) that will be applied to the reading to convert to engineering units and **soooo** is the offset that will be applied to the gage reading. The zero reading, gage factor and offset can be entered with a sign and decimal point. The maximum number of digits, including sign and decimal point is 15. The entered value will display to a maximum of 5 places to the right of the decimal point.

For all vibrating wire instruments (Gage Types 0-5), manufactured after November 2, 2011 and for all dataloggers (8002-1-X) with a firmware revision of 5.2.X and up, the basic formula for calculation of displayed and stored values is as follows;

$$\text{Display} = ((\text{CurrentReading} - \text{ZeroReading}) \times \text{Multiplier}) + \text{Offset}$$

##### Equation 1 - Displayed Gage Reading using Linear Conversion

**NOTE:** It is possible that a new datalogger might be used with an older sensor or vice versa and because of equation differences the output might be negative. As of LogView version V2.1.1.X, an additional sensor configuration parameter is now required, allowing LogView to compensate for old versus new sensor/datalogger combinations. This new parameter: **Output Calculation**, determines whether the sensor was calibrated using the formula: **G x (R0 – R1)** or **G x (R1 – R0)**. As with the gage factor, this information is available from the calibration certificate supplied with each sensor. Please see the [LogView User's Guide](#) or the online help section, "Sensor Settings" available while running LogView.

NOTE: In Equation 1 (above), the "CurrentReading" is frequently referred to as **R1** while the "ZeroReading" is referred to as **R0**

### Polynomial Conversion

The command is described further as follows; **nn** represents the gage type, or the configuration of the datalogger's input channel (see Tables 1&2 in this section), **saaaa** represents polynomial coefficient A, **sbbbb** represents polynomial coefficient B and **scccc** polynomial coefficient C. The polynomial coefficients can be entered with a sign and decimal point. The maximum number of digits, including sign and decimal point is 15. The entered value will display to a maximum of 5 places to the right of the decimal point.

For the vibrating wire instruments (Gage Types 0-5), the basic formula for calculation of displayed and stored values is as follows;

$$\text{Display} = (\text{CurrentReading}^2 \times A) + (\text{CurrentReading} \times B) + C$$

#### Equation 2 - Displayed Gage Reading using Polynomial Conversion

NOTE: When using the Polynomial conversion method, the default reading units for a vibrating wire instrument is the frequency squared multiplied by  $10^{-6}$ . For example, an instrument reading 3000Hz will output a value of "9.000" when A is entered as "0", B is "1" and C is "0". However, typical calibration units for vibrating wire instruments are frequency squared multiplied by  $10^{-3}$ . To adjust for this discrepancy between LC-2 expected units and calibration units **multiply the A coefficient by 1,000,000 and the B coefficient by 1000. The A and B coefficients can be found on the supplied calibration certificate.** The C coefficient should be calculated based on an actual field reading. The above multiplication is **only** necessary if the datalogger is being set up via text commands (see section 3.3) **AND** the gage conversion is set for polynomial. When using LogView to set up the datalogger configuration, the multiplication is performed by LogView. Refer to the appropriate sensor manual for more information on how to calculate the C coefficient.

Geokon Model	Gage Type	Description
4000	3	Strain Gage
4100	1	Strain Gage
4200	3	Strain Gage
4210	1	Strain Gage
4300BX	1	BX Borehole Stressmeter
4300EX	5	EX Borehole Stressmeter
4300NX	1	NX Borehole Stressmeter
4400	1	Embedment Jointmeter
4420	1	Crackmeter
4450	1	Displacement Transducer
4500	1	Piezometer
4600/4651/4675	1	Settlement Systems
4700	1	Temperature Transducer
4800	1	Pressure Cell
4850	1	Low Pressure Piezometer
4900	1	Load Cell
4910/4911/4912	1	Load Bolts

**Table 2 - Vibrating Wire Gage Types**

Type	Measurement Type	Description	Output Units	Linear Range	Polynomial Range
0	Vibrating Wire	Test frequency sweep, 400-4500 Hz	Digits	160 to 20250	0.160 to 20.250
1	Vibrating Wire	Middle frequency sweep, 1400-3500 Hz	Digits	1960 to 12250	1.960 to 12.250
2	Vibrating Wire	High frequency sweep, 2800-4500 Hz	Digits	7840 to 20250	7.840 to 20.250
3	Vibrating Wire	Very low frequency sweep, 400-1200 Hz	Digits	160 to 1440	0.160 to 1.440
4	Vibrating Wire	Low frequency sweep, 1200-2800 Hz	Digits	1440 to 7840	1.440 to 7.840
5	Vibrating Wire	Very high frequency sweep, 2500-4500 Hz	Digits	6250 to 20250	6.250 to 20.250
6-84	Not assigned				
85	External thermistor	Reads the thermistor encapsulated in the Vibrating Wire instrument.	°C	-50 to +80	-50 to +80
86	Internal thermistor	Reads the thermistor installed in the LC-2 Printed Circuit Board (PCB)	°C	-50 to +80	-50 to +80
87	Main battery: 12V	Reads the main 12V battery voltage	VDC	0 to 15	0 to 15
88-94	Not assigned				
95	3V lithium battery	Reads the 3V lithium RTC battery	VDC	0 to 3.5	0 to 3.5
97	Main battery: 3V	Reads the main 3V battery voltage	VDC	0 to 7.5	0 to 3.5

**Table 3 - Gage Type Descriptions**

The "Digits" calculation for the Vibrating Wire transducer output when using linear conversion is based on this equation;

$$\text{Digits} = \text{frequency}^2 \times 10^{-3}$$

**Equation 3 - Digits Calculation using Linear Conversion**

The "Digits" calculation for the Vibrating Wire transducer output when using polynomial conversion is based on this equation;

$$\text{Digits} = \text{frequency}^2 \times 10^{-6}$$

**Equation 4 - Digits Calculation using Polynomial Conversion**

Frequency, in the above equations, represents the resonant frequency of vibration of the wire in the transducer (in Hertz) as determined by the datalogger.

To convert calibration factors (pressure transducers are usually psi per digit) to other engineering units consult the following Table.

From → To ↓	psi	"H <sub>2</sub> O	'H <sub>2</sub> O	mm H <sub>2</sub> O	m H <sub>2</sub> O	"HG	mm HG	atm	mbar	bar	kPa	MPa
psi	1	.036127	.43275	.0014223	1.4223	.49116	.019337	14.696	.014503	14.5039	.14503	145.03
"H <sub>2</sub> O	27.730	1	12	.039372	39.372	13.596	.53525	406.78	.40147	401.47	4.0147	4016.1
'H <sub>2</sub> O	2.3108	.08333	1	.003281	3.281	1.133	.044604	33.8983	.033456	33.4558	.3346	334.6
mm H <sub>2</sub> O	704.32	25.399	304.788	1	1000	345.32	13.595	10332	10.197	10197	101.97	101970
m H <sub>2</sub> O	.70432	.025399	.304788	.001	1	.34532	.013595	10.332	.010197	10.197	.10197	101.97
"HG	2.036	.073552	.882624	.0028959	2.8959	1	.03937	29.920	.029529	29.529	.2953	295.3
mm HG	51.706	1.8683	22.4196	.073558	73.558	25.4	1	760	.75008	750.08	7.5008	7500.8
atm	.06805	.0024583	.0294996	.0000968	.0968	.03342	.0013158	1	.0009869	.98692	.009869	9.869
mbar	68.947	2.4908	29.8896	.098068	98.068	33.863	1.3332	1013.2	1	1000	10	10000
bar	.068947	.0024908	.0298896	.0000981	.098068	.033863	.001333	1.0132	.001	1	.01	10
kPa	6.8947	.24908	2.98896	.0098068	9.8068	3.3863	.13332	101.320	.1	100	1	1000
MPa	.006895	.000249	.002988	.00000981	.009807	.003386	.000133	.101320	.0001	.1	.001	1

**Table 4 - Engineering Units Multiplication Factors**

#### 4.9. IDdddddddddddddddddd

Displays or sets the datalogger ID. The ID is a 16 character string that can be used to identify a datalogger and the data that is transmitted by it. If an ID is entered it will be transmitted as the first element in each array of data. For example;

```
*ID
Datalogger ID:
*IDDatalogger#1
Datalogger ID:Datalogger#1
*ST
Logging started.
Datalogger#1,2009,94,0939,20,3.22,23.19,9986.034,25.0,1

*Datalogger#1,2009,94,0939,25,3.22,23.36,9985.864,25.0,2
Datalogger#1,2009,94,0939,30,3.22,23.44,9985.479,25.0,3
Datalogger#1,2009,94,0939,35,3.22,23.53,9985.686,25.0,4
```

To clear the ID enter a <SPACE> character as the ID. When the ID is cleared the arrays from the logger will display beginning with the year. To display the current ID enter **ID** <ENTER>.

#### 4.10. LC

Selects the linear conversion method for the instrument reading. See the G command section for more information.

```
*LC
Linear conversion selected.
*
```

#### 4.11. L

Display all 6 log intervals.

```
*L
                        Log Intervals List
-----
Interval #1 Length: 10   Iterations: 100
Interval #2 Length: 20   Iterations: 90
Interval #3 Length: 30   Iterations: 80
Interval #4 Length: 40   Iterations: 70
Interval #5 Length: 50   Iterations: 60
Interval #6 Length: 60   Iterations: 0
*
```

This command has no effect on the current interval (scan or log). If logging is started and log intervals are enabled the iterations value will be followed by the number of readings left at that interval. For example;

\*L

**Log Intervals List****Interval #1 Length: 10 Iterations: 100/58****Interval #2 Length: 20 Iterations: 90/90****Interval #3 Length: 30 Iterations: 80/80****Interval #4 Length: 40 Iterations: 70/70****Interval #5 Length: 50 Iterations: 60/60****Interval #6 Length: 60 Iterations: 0/0**

\*

The above list indicates that there are 42 iterations of interval 1 left before interval 2 begins execution. See the **Ln/11111/iii** command section to modify intervals.

**4.12. Ln/IIII/iii**

Define the length and iteration of any interval in the list; n refers to the number of the interval (1-6), IIII is the length (3-86400), and iii is the iterations (0-255), or the number of readings that will be taken at that interval. If 0 is entered for the iteration value that interval will execute indefinitely. Illegal entries will be ignored, i.e. **L7/10/100** or **L1/1000/500**. If the entry is correct the modified interval will display.

\*L1/100/0

**Interval #1 Length: 100 Iterations: 0**

\*

**If log intervals are enabled and logging was started, any change to the interval list will result in a restart of the table!**

Table 5 lists possible logarithmic interval lengths and iterations. Any combination of lengths and iterations is permissible.

<b>Interval</b>	<b>Length</b>	<b>Iterations</b>	<b>Elapsed Time</b>
1	3 seconds	3	.1 minute
2	6 seconds	9	1 minute
3	10 seconds	54	10 minutes
4	30 seconds	180	100 minutes
5	240 seconds	225	1000 minutes
6	3600 seconds	endless	

**Table 5 - Logarithmic Intervals List**

#### 4.13. LD

Disable use of log intervals. If logging is started (**ST** command) it will continue based on the scan interval entry (**SC** command).

```
*LD
Log intervals disabled.
*Datalogger#1,2009,94,1002,46,3.22,24.39,9986.389,25.0,1
*
```

#### 4.14. LE

Enable use of log intervals. If logging is started (**ST** command) it will continue based on the interval lengths and iterations of the log list (**SC** command).

```
*LE
Log intervals enabled.
*Datalogger#1,2009,94,1004,35,3.22,23.36,9986.394,25.0,1
*
```

#### 4.15. M

Display the current Monitor mode setting. The monitor mode will display arrays as they are stored in memory in the course of logging. This is useful where a test is being conducted and immediate display of logged values would be helpful. Use the **MD** and **ME** commands (next two sections) to disable or enable the use of the Monitor mode.

```
*M
Monitor mode enabled.
*
```

#### 4.16. MD

Disable the Monitor mode. Arrays will not be sent to the host computer as they are logged.

```
*MD
Monitor mode disabled.
*
```

#### 4.17. ME

Enable the Monitor mode. Arrays will be sent to the host computer as they are logged.

```
*ME
Monitor mode enabled.
*
```

#### 4.18. MS

Display the current Memory Status. Maximum number of readings (8000 or 16000) will be displayed, along with the wrap format and status of reading synchronization.

```
*MS
8000 readings maximum
Logging will not stop when memory is full
Readings are synchronized to the top of the hour.
*
```

#### 4.19. N

Display the next time the datalogger will initiate a measurement cycle. If the start time (**ST** command) has been set this command will display when logging will begin.

```
*ST10:48
Logging will start at: 10:48:00
*N
Next time to read: 10:48:00
*
```

#### 4.20. NA

Displays the current network address.

```
*NA
Network address: 1
*
```

When network recognition is enabled, this number (preceded by the # character) must be entered for the respective datalogger to respond. The following example illustrates communication with 2 different dataloggers on the RS-485 network.

```
<ENTER>
<ENTER>
#1<ENTER>
Network address: 1
*NA
Network address: 1
*E

<ENTER>
<ENTER>
#2<ENTER>
Network address: 2
*NA
Network address: 2
*E
```



## 4.21. NAddd

Sets the current network address to any address between 1 and 256.

```
*NA10
Network address: 10
*
```

When network recognition is enabled, this number (preceded by the # character) must be entered for the respective datalogger to respond. The datalogger then responds by transmitting its network address. The following example illustrates communication with 2 different dataloggers on the RS-485 network.

```
<ENTER>
<ENTER>
#1<ENTER>
Network address: 1
*E

<ENTER>
<ENTER>
#20<ENTER>
Network address: 20
*E
```

NOTE: If connected directly to the datalogger via USB and networking is enabled, the datalogger will respond with the \* prompt only.

NOTE: The network address may not be changed while networked. Direct connect to the datalogger via USB in order to change the network address.

## 4.22. ND

Network Disable the datalogger. Disables networking of 2 or more LC-2 dataloggers.

```
*ND
Network recognition disabled.
```

NOTE: Networking may not be disabled while networked. Direct connect to the datalogger via USB in order to disable networking.

## 4.23. NE

Network Enable the datalogger. Enables networking of 2 or more LC-2 dataloggers.

```
*NE
Network recognition enabled.
```

Note: If the LC-2 is connected via the USB port, connection to a network enabled datalogger can be made directly without the need to enter the correct datalogger address. This can be helpful if the network address is unknown and the datalogger is network enabled.

#### 4.24. NS

Display the current network status.

```
*NS
Network recognition disabled.
*
```

Or;

```
*NS
Network recognition enabled.
*
```

#### 4.25. PC

Selects the Polynomial Conversion method for the instrument reading. See the **G** command section for more information.

```
*PC
Polynomial conversion selected.
*
```

#### 4.26. Pnnnn

Position the User Position memory pointer. Type **P** and a number between 1 and 8000 (or 1 and 16000 if 16000 readings is enabled) to position the pointer. Arrays can then be displayed (**D** command) from the new position. The updated pointers will display after entering a valid position.

```
*P1
MS:3200   OP:1567   UP:1
*
```

#### 4.27. R

Reset memory pointers to default settings. Gage and interval settings, as well as the real-time clock settings, are not affected by this command. User will be asked to verify before executing. Press **Y** to continue, any other key to abort.

```
*R
Are you sure(Y/N)?Y
Memory cleared.
*
```

Note: This command does not erase memory. If the need arises to recover data that was previously taken, take 1 (or more) readings and then position the memory pointers via the **P** and **D** commands to recover previously taken readings.

#### 4.28. RESET

**RESET** (re-boot) the LC-2 microprocessor. All stored readings and settings, as well as the ID and real-time clock settings are not affected by this command.

```
*RESET
Resetting...
RESET COMPLETE
*
```

#### 4.29. RT

**Readings Total.** Displays the total number of readings that the datalogger will take (8000 or 16000) before either overwriting data memory or stopping logging (depending on the Wrap Format status).

```
*RT
8000 readings maximum
*
```

#### 4.30. SR

**Synchronize Readings.** Displays status of reading synchronization

```
*SR
Readings are synchronized to the top of the hour.
*
```

#### 4.31. SR0

Readings will not be synchronized to the top of the hour. All subsequent readings will occur at the time of the first reading plus the scan interval.

```
*SR0
Readings are not synchronized to the top of the hour.
*SC5
Scan interval: 5 second(s).
*ST
Logging started.
2009,195,0921,28,3.07,24.05,-9025.851,23.4,1

*2009,195,0921,33,3.07,24.10,-9025.595,23.4,2

*2009,195,0921,38,3.07,24.16,-9025.825,23.4,3
2009,195,0921,43,3.07,24.16,-9025.317,23.4,4
2009,195,0921,48,3.07,24.16,-9025.618,23.4,5
2009,195,0921,53,3.07,24.13,-9025.377,23.4,6
```

#### 4.32. SR1

(Default) Readings will be synchronized to the top of the hour. All subsequent readings will occur at the specified scan interval while evenly divisible into the top of the hour.

```
*SR1
Readings are synchronized to the top of the hour.
*SC5
Scan interval: 5 second(s).
*ST
Logging started.
2009,195,0923,17,3.07,23.93,-9025.767,23.4,1

*2009,195,0923,20,3.07,23.96,-9025.185,23.4,2

*2009,195,0923,25,3.07,24.05,-9025.486,23.4,3
2009,195,0923,30,3.07,24.08,-9025.754,23.4,4
2009,195,0923,35,3.07,24.08,-9025.632,23.4,5
2009,195,0923,40,3.07,24.08,-9025.486,23.4,6
```

#### 4.33. RT8

Sets the Total number of Readings to 8000.

```
*RT8
8000 readings maximum
*
```

#### 4.34. RT16

Sets the Total number of Readings to 16000.

```
*RT16
16000 readings maximum
*
```

#### 4.35. S

Display the datalogger Status.

```
*S
MS:3200 OP:1567 UP:1
GT: 1 GZ: 8934.0000 GF: 0.01234 GO: 0.00000
Scan interval: 60 second(s).
Logging started.
Logging will stop at: 10:50:00
Log intervals enabled.
Monitor mode disabled.
*
```

Line	Description	Manual Sections
1	Status of memory pointers	4.4, 4.24
2	Gage information	4.6
3	Scan interval setting	4.34
4	Start/Stop status	4.33, 4.36, 4.37
5	Stop time (optional)	4.36
6	Log interval status	4.11, 4.12
7	Monitor mode status	4.13

**Table 6 - S Command Information**

#### 4.36. SCnnnnn

Enter the Scan interval, in seconds. Range of entry is 3 to 86400. Only whole numbers are accepted. Typing **SC** with no value returns the current setting only

```
*SC
Scan interval: 60 second(s).
*SC300
Scan interval: 300 second(s).

*
```

#### 4.37. SS

Display the System Status of the datalogger.

```
*SS
Signature of RAM1: 32819
Signature of RAM2: 15979
Signature of RAM3: 63255
Signature of RAM4: 2197
Signature of ROM: 15283
Trap count: 0
Network address: 1
Network recognition disabled.
Time format is hhmm.
Date format is julian.

*
```

Line	Description
1	Signature of RAM bank 1. (checksum)
2	Signature of RAM bank 2. (checksum)
3	Signature of RAM bank 3. (checksum)
4	Signature of RAM bank 4. (checksum)
5	Signature of ROM (checksum)
6	Communication errors counter.
7	Current network address.
8	Current network status.
9	Current time format configuration.
10	Current date format configuration.

**Table 7 - SS Command Information**

#### 4.38. SPhh:mm

**StoP** the datalogger logging values; hh is the hour (24 hour format) of the day to stop and mm the minutes. The time entry is optional.

```
*SC60
Scan interval: 60 second(s).
*ST
Logging started.
2009,92,1512,46,3.24,25.66,12046.43,22.33,1

*SP12:00
Logging will start at: 15:13:46
Logging will stop at: 12:00:00
*
```

Note that when SPhh:mm is issued, the datalogger responds with the time of the next reading along with the time at which logging will stop.

#### 4.39. SThh:mm

**ST**art the datalogger logging values; **hh** is the hour of the day (24 hour format) to start and **mm** the minutes. The time entry is optional. Entry is ignored if logging is already started (unless a time is entered).

```
*ST
Logging already started!
*ST11:00
Logging will start at: 11:00:00
Logging will stop at: 12:00:00
*
```

#### 4.40. SV

Return the **Software Version** of the datalogger's operating system software. Consult the factory to check on latest versions available.

```
*SV
Software version: 4.17.0
*
```

#### 4.41. TEST

**TEST** is a set of internal self tests that are performed at the factory during final test.

\*TEST

LC-2 TEST MENU:

SELECTION      TEST

- 0    INTERNAL EEPROM
- 1    EXTERNAL EEPROM BANK 1
- 2    EXTERNAL EEPROM BANK 2
- 3    EXTERNAL EEPROM BANK 3
- 4    EXTERNAL EEPROM BANK 4
- 5    EXTERNAL EEPROM BANK 5
- 6    EXTERNAL EEPROM BANK 6
- 7    ALL EEPROM
- 8    +5X\_X
- 9    RTC 32KHz
- A    EXTERNAL INPUT (GAGE TYPE 1)
- B    EXTERNAL INPUT (GAGE TYPE 2)
- C    EXTERNAL INPUT (GAGE TYPE 3)
- D    EXTERNAL INPUT (GAGE TYPE 4)
- E    EXTERNAL INPUT (GAGE TYPE 5)

ENTER SELECTION:

Selection	Description
0	Test the Configuration memory bank
1	Test Readings 1-3200 memory bank
2	Test Readings 3201-6400 memory bank
3	Test Readings 6401-9600 memory bank
4	Test Readings 9601-12800 memory bank
5	Test Readings 12801-16000 memory bank
6	Test Readings 16001-19200 memory bank
7	Test all memory banks
8	Turn on system power supplies
9	Test the 32.768 RTC timebase
A	External test input configuration: gage type 1
B	External test input configuration: gage type 2
C	External test input configuration: gage type 3
D	External test input configuration: gage type 4
E	External test input configuration: gage type 5
X	Exit and return to normal operations

**Table 8 – TEST Menu Information**

#### 4.42. TF

Display the current **T**ime **F**ormat display option setting. This setting determines how the time information will be displayed in the array when the Monitor mode is active (see **M** command section) or arrays are being displayed from memory. Entering TF alone returns the current time format. Entering TF0 sets the time format to hhmm. Entering TF1 sets the time format to hh,mm. The default time format display is hhmm.

```
*TF0
Time format is hhmm.
*D
2009,52,1343,30,3.00,24.7,-5372.293,24.3,57
*TF1
Time format is hh,mm.
*D
2009,52,13,43,30,3.00,24.7,-5372.293,24.3,57
*
```

#### 4.43. TR

Display the current **T**Rap Count. The trap counter is a register that keeps track of the number of times that the internal processor has detected a communications error. This is a useful register to check if communication problems are suspected.

#### 4.44. TR0

Reset the **T**Rap count register to **0**.

#### 4.45. WF

Display the current Wrap Format. Memory “wrapping” means that once the memory has filled, the datalogger will continue taking readings and overwrite the stored values in a circular fashion (see section 3.4.Dnnnn).

When the wrap format is set to 0, logging will stop once the memory becomes full. This is useful if critical data is stored and it must not be inadvertently overwritten and lost.

When the wrap format is set to 1, logging will continue when the memory becomes full and the original stored values will be overwritten. With this setting, logging will continue indefinitely until told to stop with the SP command, or the programmed stop time has been reached.

```
*WF
Logging will not stop when memory is full
*WF0
Logging will stop when memory is full
*WF1
Logging will not stop when memory is full
*
```



**4.46. X**

Take and display one reading, but do not store this reading in memory. Useful if interested in obtaining a reading at the moment, without interrupting or affecting the current logging schedule. The User Position is not displayed with the array data.

**\*D**

**There are no arrays to display.**

**\*X**

**2009,92,1603,12,3.00,24.7,-5372.293,24.3**

**\*D**

**There are no arrays to display.**

**\***

## **5. MAINTENANCE**

Although Model LC-2 Datalogger is designed to operate in field environments, nevertheless there are some basic maintenance procedures that should be followed to insure maximum reliability and functionality.

### **5.1. Keeping the Inside of the box dry:**

The LC-2 datalogger is designed to be splash proof and rain proof but is not designed to be submersible under water. The LC-2 enclosure lid is sealed by a gasket which will remain sealed so long as the lid screws are kept tight. Most important is to make sure that the Hubble connector around the cable entry is securely tightened so that the internal grommet grips and seals around the cable. LC-2 models that use a 10 pin connector as a cable entry are equipped with sealing caps which must be kept tightened to the connector when the connector is not in use.

Despite all these precautions the LC-2 may encounter leakage along the cable if the cable is cut, and/or condensation problems especially in humid environments. In these environments it is recommended that the internal desiccant pack be replaced at the necessary intervals to prevent condensation from corroding or shorting out the internal electronics.

### **5.2. Cleaning:**

The outside of the box can be cleaned using a cloth dampened with soap and water. **DO NOT USE ANY TYPES OF SOLVENTS OR SCOURING AGENTS!**

The connector sockets can be cleaned using a small stiff brush (small painters brush) dipped in soap and water. The sockets are water resistant so the internal electronics will not be adversely affected by them filling with water or other liquids. Be aware however, readings could be affected by shorting or other effects of an improper connection due to fluids being present in the connector. Dry connections thoroughly before using.

### **5.3. Batteries:**

When the unit is not in use, especially for extended periods of time, the batteries should be removed to prevent damage due to leakage. The warranty does not cover damage due to battery leakage. The table below details approximate operating times for the various types of 'D' cell batteries that may be used with the Model LC-2.

Battery Chemistry (2 D-cells)	Battery Pack Voltage	Battery Capacity	2 Second Scan Rate	1 Minute Scan Rate	1 Hour Scan Rate	1 Day Scan Rate
Lithium	7.2V	19 AHr	10.4 days	613 days	≥2 years	≥2 years
Alkaline	3.0V	14 AHr	3.2 days	188 days	≥2 years	≥2 years
Carbon-Zinc	3.0V	5 AHr	1.1 day	66.2 days	≥1 year	≥1 year

**Table 9 - Approximate Operating Times**

The above table assumes a constant temperature environment of 25°C (not field conditions!). Battery life is shortened by temperature extremes. For models 8002-1-2 and 8002-1A-2, if the datalogger is continuously connected to an active computers USB port, all operating power will be supplied via the USB port. As soon as USB power is lost, the datalogger will immediately switch over to its internal 3V (or external 12V) battery pack.

Batteries should be replaced when the measured voltage drops below 1.8 VDC (internal D-cells) or 6V (external 12V battery). The datalogger electronics will stop the datalogger from logging and disable RS-485 communications if the battery goes below 1.6 VDC (internal D-cells) or 5.5V (external 12V battery). In this event, a new set of batteries must be installed before the datalogger becomes operable again. All data and operating parameters are retained when removing batteries, even for an extended period (years) of time due to non-volatile EEPROM memory. If the datalogger was logging when it stopped itself due to low battery voltage, it will resume logging as soon as new batteries are installed.

**Battery replacement instructions:**

- 1) Remove the 4 captive lock regular head screws on the top of the case and lift the cover off. Underneath the cover is the 'D' cell battery holder.
- 2) Remove the two batteries from the holder being careful not to bend the sides outward. Note the polarity outline on the bottom of the battery holder for proper battery installation. Insert the new batteries straight down into the holder. Check for secure connection between the battery terminals and holder. If a gap exists, remove batteries and bend the holder sides inward.



Proper Battery installation



Faulty Battery installation

- 3) Re-install the cover. Check datalogger for proper operation.

## **6. TROUBLESHOOTING**

Listed below are a few commonly experienced problems and remedial action. Contact the factory should a problem arise not explained herein or additional information be needed.

### **6.1. Unit will not respond to communications.**

- ✓ Wrong COM port selected in LogView. See LogView manual
- ✓ If RS-232 or RS-485 communications are being used, the internal batteries of the datalogger may be low, dead or have a faulty connection to the holder. Replace/check the batteries according to the **“Battery replacement instructions:”** on the previous page.
- ✓ If RS-485 communications is being used, the <ENTER>,<ENTER>, #, **datalogger address**, <ENTER> key sequence is not being sent. Refer to Appendix F – NETWORKING for further information.

### **6.2. Vibrating wire gage measurement reads -999999.0**

- ✓ Using an ohmmeter, check connections to the vibrating wire gage leads. Resistance should be between 90 and 180 ohms (pins A&B on the 10-pin connector, see Appendix B). Remember to correct for cable resistance (approximately 15  $\Omega$ /1000' or 50  $\Omega$ /km, double for both directions). If resistance reads less than 100  $\Omega$  the cable is probably shorted. If resistance reads infinite or in the megohms range the cable is probably cut.
- ✓ Check the datalogger with another gage. If it reads okay, the datalogger may be malfunctioning.
- ✓ Check that the proper gage type is selected and connected properly (see TABLE 1, TRANSDUCER CABLE CONNECTIONS and TABLE 3, GAGE TYPE LISTING).

### **6.3. Gage measurement (analog or vibrating wire) reads -999999.9**

- ✓ A mathematical over-range has occurred. Check the magnitude of the reading, zero reading, multiplier and offset. The result must be in the range of  $1.0 \times 10^{-7}$  to  $1.0 \times 10^7$ .

### **6.4. Vibrating wire gage reading is unstable**

- ✓ Is there a source of electrical noise nearby? Likely candidates are generators, motors, arc welding equipment, high voltage lines, etc. If possible, move the datalogger and transducer cable away from the power lines or electrical equipment.
- ✓ Check if the proper gage type is selected (see TABLE 1, TRANSDUCER CABLE CONNECTIONS and TABLE 3, GAGE TYPE LISTING).

### **6.5. Thermistor measurement shows -99.9 degrees Celsius**

- ✓ Indicates open circuit to thermistor leads. Check connections from datalogger to thermistor leads. If okay, check thermistor with ohmmeter. APPENDIX E, THERMISTOR TEMPERATURE DERIVATION details the resistance versus temperature relationship. It should read between 10K ohms and 2.4K ohms (0° to +30° Celsius). If thermistor checks out okay consult the factory to schedule repair of unit.

## **APPENDIX A - SPECIFICATIONS**

### **A.1. Measurement Capability**

- Vibrating Wire (all types).
- External temperature (thermistor).
- Internal temperature (thermistor).
- Main battery voltage.(3V and 12V)
- RTC lithium battery voltage.

### **A.2. Power**

Power supply:	Internal 3 VDC (7.5Vmax) or External 12 VDC (15Vmax)
Processing/communication current:	<100 mA
VW measurement current:	<250 mA
Quiescent current:	<600 $\mu$ A
RTC battery type:	Panasonic CR2032 3V lithium coin cell: 20mm, 225 mAHr
RTC battery life:	>10 years
Operating temperature range:	-30 to +50° C

### **A.3. Memory**

Data memory:	320K EEPROM
Program memory:	24K EEPROM
Array storage	8000 or 16000
Data memory type:	ring (oldest over-write)
Array elements:	ID (optional) Year Julian day (or month,day) Time (hhmm or hh,mm) Seconds Battery voltage Datalogger temperature Transducer reading Transducer temperature Array #

### **A.4. Clock**

Features:	full calendar
Time format:	12 or 24 hour (selectable)
Date Format:	mm,dd or julian (selectable)
Accuracy:	$\pm$ 2 minutes per year

**A.5. Serial Interface (all LC-2 models):**

Speed: 9600 & 115,200 bps (version 5.2.X and later)  
 Parameters: 8 Data bits  
                   1 Stop bit  
                   No Parity  
                   No Flow Control  
 Data output format: ASCII text

**A.6. RS-485 Network**

Maximum nodes: 256  
 Maximum cable length: 4000', 1.22 km

**A.7. Vibrating Wire Measurement**

Excitation sweep range: 400 Hz to 4500 Hz  
 Frequency Measurement Technique: Adaptive Multiple Period Averaging  
 Accuracy: 0.05% F.S.R. (450-4000 Hz)  
 Resolution: 0.001 digit

**A.8. Internal/External Temperature Measurement**

Thermistor: Dale #1C3001-B3 (YSI 44005)  
 Transducer accuracy:  $\pm 0.5^{\circ}\text{C}$   
 Measurement accuracy: 0.5% FSR  
 Resolution:  $0.01^{\circ}\text{C}$  (Internal)  
                    $0.1^{\circ}\text{C}$  (External)  
 Linearization error: 0.02% FSR  
 Temperature range:  $-40$  to  $+60^{\circ}\text{C}$   
 Overall accuracy: 1.0% FSR ( $\pm 1^{\circ}$ )

**A.9. Main Battery Measurement**

<u>3V Battery:</u>		<u>12V Battery:</u>	
Range:	0 to 7.5 VDC	Range:	0 to 15 VDC
Accuracy:	$\pm 1.83\text{mV}$	Accuracy:	$\pm 3.662\text{mV}$
Resolution:	0.01 VDC	Resolution:	0.01 VD

## **APPENDIX B - CONNECTOR PINOUTS**

### **B.1 Transducer Connections**

#### **B.1.1 Transducer Cable Connections (8002-1-2)**

<b>Terminal Strip Position</b>	<b>Internal Wire Color</b>	<b>PCB connector J7 pin</b>	<b>Description</b>	<b>Cable Wire Color</b>
VW+	Brown	1	Vibrating Wire +	RED
VW-	Red	2	Vibrating Wire -	BLACK
TH+	Orange	3	Thermistor +	GREEN
TH-	Yellow	4	Thermistor -	WHITE
S	Green	5	Analog Ground (shields)	BARE WIRE

**Table B-1A Transducer Cable Connections (USB Datalogger)**

#### **B.1.1.2 Transducer Cable Connections (8002-1-1)**

<b>Terminal Strip Position</b>	<b>Internal Wire Color</b>	<b>PCB connector J6 pin</b>	<b>Description</b>	<b>Cable Wire Color</b>
VW+	Brown	1	Vibrating Wire +	RED
VW-	Red	2	Vibrating Wire -	BLACK
TH+	Orange	3	Thermistor +	GREEN
TH-	Yellow	4	Thermistor -	WHITE
S	Green	5	Analog Ground (shields)	BARE WIRE

**Table B-1B Transducer Cable Connections (Serial Datalogger)**

### **B.2. Sensor Connector Pin-out (8002-1A-1, 8002-2A-1)**

The mating 10 pin Bendix plug is part number PT06F-12-10P.

<b>10 Pin Bendix</b>	<b>Inside Color</b>	<b>Description</b>	<b>Transducer Wire Color</b>
A	Brown	Vibrating Wire +	Red
B	Red	Vibrating Wire -	Black
C	Orange	Thermistor +	Green
D	Yellow	Thermistor -	White
E	Green	Analog Ground (shields)	Shield
F	Blue	+5VDC Supply (switched)	N/A
G	Violet	Digital Ground	N/A
H	Grey	Mux Reset	N/A
J	White	Mux Clock	N/A
K	Black	Digital Ground	N/A

**Table B-2 Sensor Connector Pin-out**

### B.3. COM Connector Pin-out

The mating 10 pin Bendix plug is part number PT06F-12-10P.

10 Pin Bendix	Internal Wire Color	PCB connector J5 pin	Description	
			USB	RS-232
A	Brown	1	USB VCC	Digital Ground
B	Red	2	USB DM	Tx
C	Orange	3	USB DP	Rx
D	Yellow	4	Digital Ground	RTS
E	Green	5	RS-485 RX	CTS
F	Blue	6	RS-485 /RX	n.c.
G	Violet	7	RS-485 TX	DTR
H	Grey	8	RS-485 /TX	+5V
J	White	9	RS-485 +12V	n.c.
K	Black	10	RS-485 Ground	Digital Ground

**Table B-3 COM Connector Pin-out**

### B.4. RS-485 Connector Pin-out (optional – 8002-1-3)

10 Pin Bendix	Internal Wire Color	PCB connector J6 pin	Description
A	Brown	1	No Connection
B	Red	2	No Connection
C	Orange	3	No Connection
D	Yellow	4	Digital Ground
E	Green	5	RS-485 RX
F	Blue	6	RS-485 /RX
G	Violet	7	RS-485 TX
H	Grey	8	RS-485 /TX
J	White	9	RS-485 +12V
K	Black	10	RS-485 Ground

**Table B-4 RS-485 Connector Pin-out**



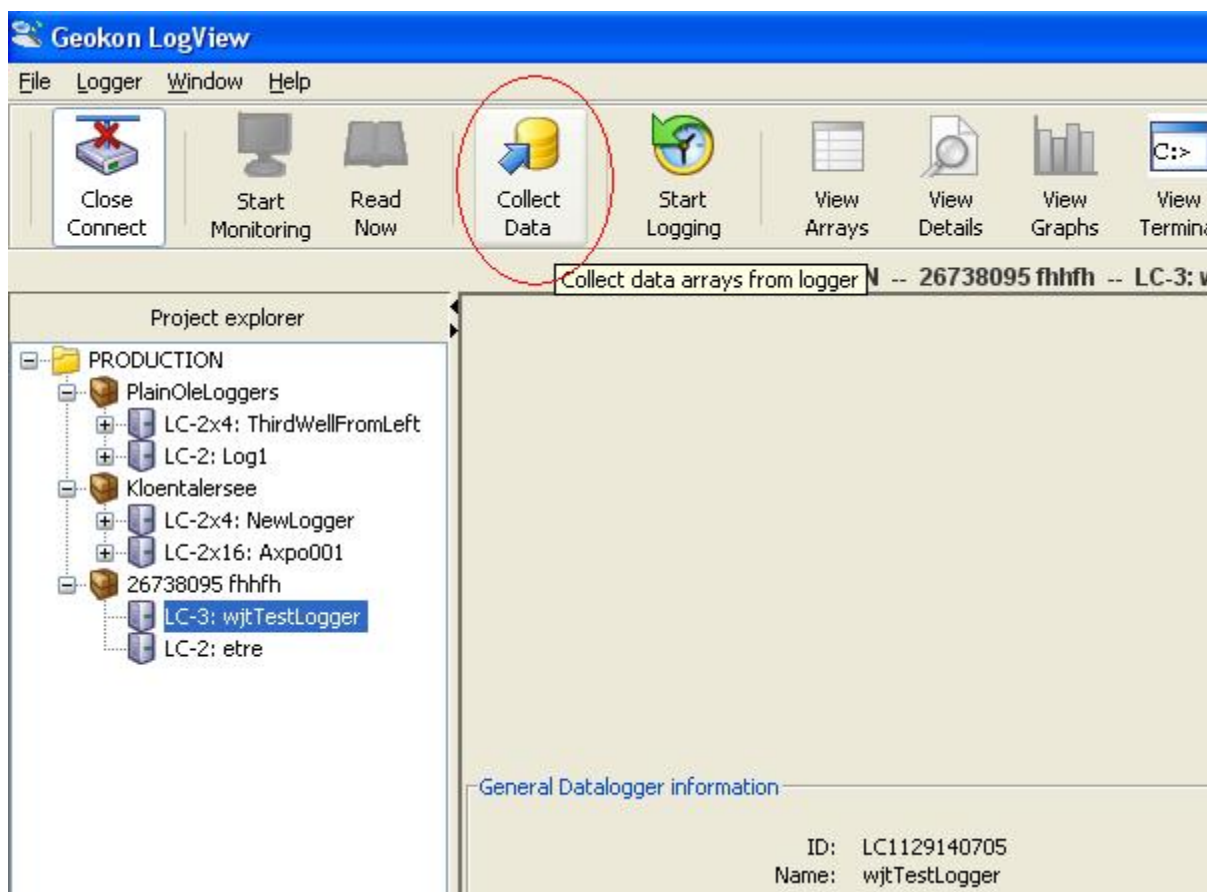
## **APPENDIX C - DATA FILE TRANSFER TO A WINDOWS PC**

Data can be downloaded to the PC either via LogView software (refer to the LogView Online Help) or Windows HyperTerminal, which, prior to Windows Vista, was supplied with most personal computers. The steps to download the data using LogView are as follows:

### **C.1. Downloading Data using LogView**

The steps below assume that a successful connection has been previously established between LogView and the datalogger. (See section 3.2.8 of this manual)

Click on the Collect Data button from the Main Toolbar. See figure 17 below:

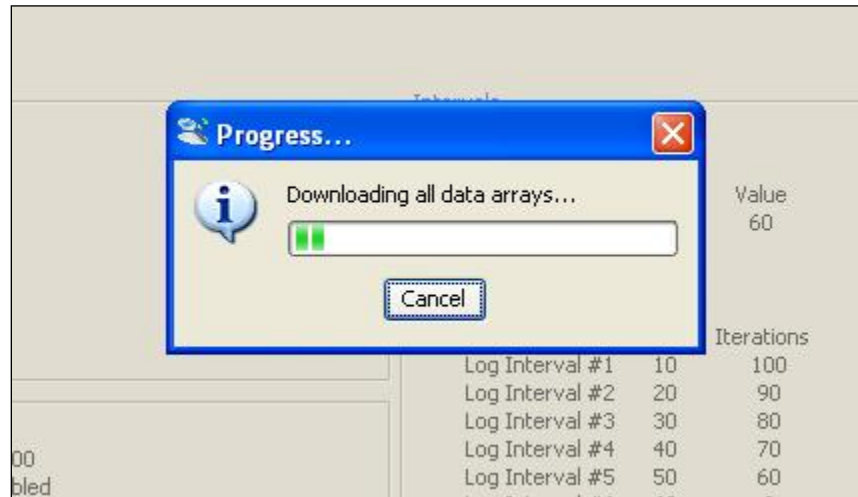


**Figure 17 - LogView Collect Data Button**

If the datalogger configuration is set for “Collect all data” in “Datalogger Settings->Data Collection Options” (see the LogView on-line help menu covering datalogger settings) then LogView will issue commands to the datalogger to initiate a download of all arrays logged on the datalogger. If the memory has wrapped then 16000 arrays (8000 if datalogger configuration set to **RT8**, see sections 4.29 and 4.33) will be downloaded starting at the current User Pointer (See sections 4.6 and 4.26 of this manual).

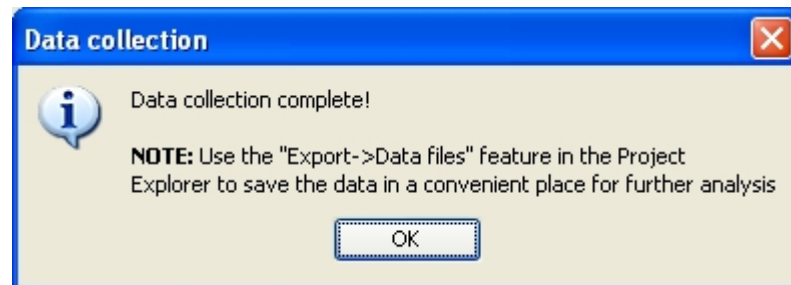
If the datalogger configuration is set for “Collect new data since last download” in “Datalogger Settings->Data Collection Options” then LogView will issue commands to the datalogger to initiate a download of all arrays since the last time data was downloaded.

Once the data collection has been initiated, the following progress bar (see figure 18) will be displayed until the collection has completed:



**Figure 18 - Data Collection Progress Bar**

After a data collection has finished LogView will display the message shown in figure 19:



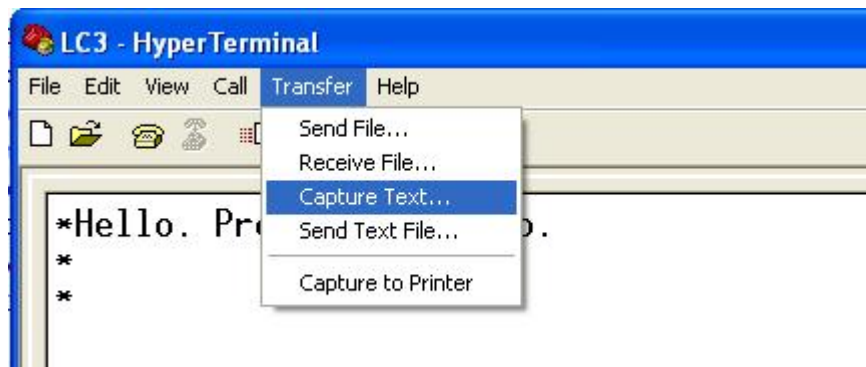
**Figure 19 - Data Collection Complete Message**

## C.2. Downloading Data using HyperTerminal (or equivalent)

The steps to download the data using HyperTerminal are as follows:

Launch HyperTerminal: Start → Programs → Accessories → Communications → HyperTerminal

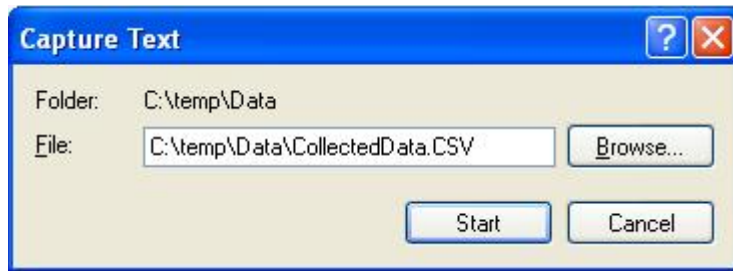
1. Enter a name for the New Connection – Select OK (see section 3.3, Figure 13, HyperTerminal Connection Description).
2. Change the “Connect using” setting to the appropriate COM port (in this case COM3 - see section 3.3, Figure 14, HyperTerminal Connection Selection).
3. In the COM Properties Dialog, enter the “Port Settings”. Select Apply. Select OK (see section 3.3, Figure 15, HyperTerminal COM Port Settings).
4. With the cursor in the display screen, press the Enter key a few times to verify that communications has been established. The datalogger should return the power up prompt;  
**Hello. Press "?" for Help.**  
\*
5. Upon confirmation of communications, select Transfer | Capture Text (see figure 20):



**Figure 20 - HyperTerminal Transfer Menu**

6. Enter the path and name of the file you wish to create, either directly or with the Browse button, then click on the Start button (see Figure 21).

**Hint:** It may be helpful to specify .CSV as the file extension to allow direct formatted entry into a spreadsheet program.



**Figure 21 - Specify Data Capture File**

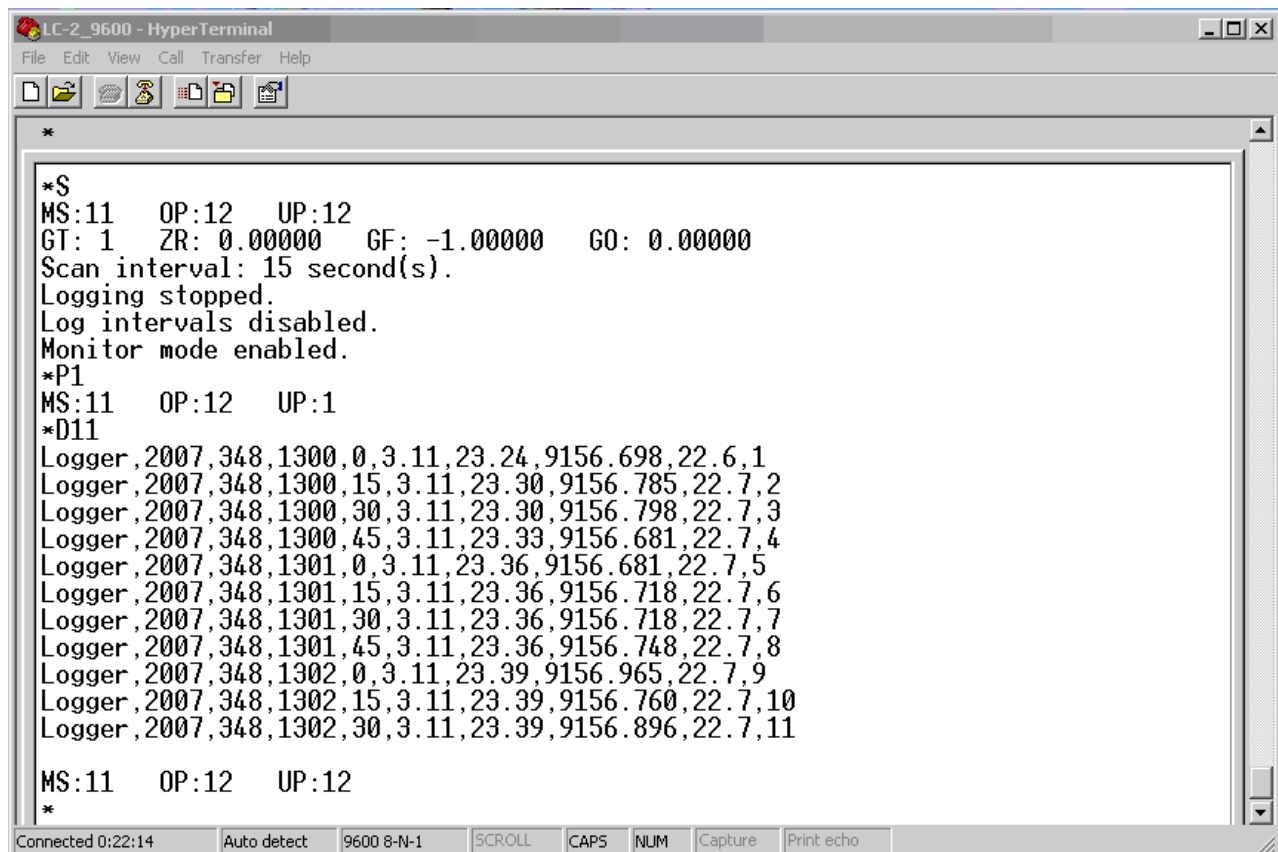
7. With the cursor in the display screen, push the <Enter> key a few times to wake-up the datalogger

Type "S" to get the Status of the datalogger.

Type "P1" to position the data array Pointer at location 1.

Type "D11" to Display the readings stored in memory (see Figure 22).

Select Transfer | Capture Text | Stop.



**Figure 22 - HyperTerminal/Datalogger Communication**

8. The data are now stored in the specified file.

## **APPENDIX D - SAMPLE DATA FILES**

### **D.1. Sample Raw Data File**

Note: the datalogger ID feature (see **ID** command) is not being used.

```

2009,94,1401,30,3.22,24.02,10010.198,25.0,1
2009,94,1401,35,3.22,24.10,10010.213,25.0,2
2009,94,1401,40,3.22,24.13,10009.919,25.0,3
2009,94,1401,45,3.22,24.22,10010.012,25.0,4
2009,94,1401,50,3.22,24.25,10010.125,25.0,5
2009,94,1401,55,3.22,24.25,10010.165,25.0,6
2009,94,1402,0,3.22,24.13,10010.205,25.0,7
2009,94,1402,5,3.22,24.05,10010.031,25.0,8
2009,94,1402,10,3.22,23.99,10010.158,25.0,9
2009,94,1402,15,3.22,23.96,10010.052,25.0,10
2009,94,1402,20,3.22,23.90,10010.140,25.0,11
2009,94,1402,25,3.22,23.87,10010.313,25.0,12
2009,94,1402,30,3.22,23.87,10009.919,25.0,13
2009,94,1402,35,3.22,23.84,10010.146,25.0,14
2009,94,1402,40,3.22,23.84,10010.454,25.0,15
2009,94,1402,45,3.22,23.84,10010.227,25.0,16
2009,94,1402,50,3.22,23.82,10010.280,25.0,17

```

Column: 1   2   3   4   5   6   7   8   9

where;

Column 1 represents the year when the array was stored.

Column 2 represents the julian day (or day, month format, see section 4.5.).

Column 3 represents the time (or hh,mm format, see section 4.42.).

Column 4 represents the seconds.

Column 5 represents the main battery voltage (alkaline batteries, nominal 3.0 VDC).

Column 6 represents the internal temperature in degrees Celsius.

Column 7 represents the datalogger measurement (as specified by the Gnn command).

Column 8 represents the external temperature in degrees Celsius.

Column 9 represents the array number.

**D.2. Sample Formatted Data File**

Year	Day	Time	Secs	Battery	D_Temp	Digits	S_Temp	Array
2009	94	1401	30	3.22	24.02	10010.198	25.0	1
2009	94	1401	35	3.22	24.10	10010.213	25.0	2
2009	94	1401	40	3.22	24.13	10009.919	25.0	3
2009	94	1401	45	3.22	24.22	10010.012	25.0	4
2009	94	1401	50	3.22	24.25	10010.125	25.0	5
2009	94	1401	55	3.22	24.25	10010.165	25.0	6
2009	94	1402	0	3.22	24.13	10010.205	25.0	7
2009	94	1402	5	3.22	24.05	10010.031	25.0	8
2009	94	1402	10	3.22	23.99	10010.158	25.0	9
2009	94	1402	15	3.22	23.96	10010.052	25.0	10
2009	94	1402	20	3.22	23.90	10010.140	25.0	11
2009	94	1402	25	3.22	23.87	10010.313	25.0	12
2009	94	1402	30	3.22	23.87	10009.919	25.0	13
2009	94	1402	35	3.22	23.84	10010.146	25.0	14
2009	94	1402	40	3.22	23.84	10010.454	25.0	15
2009	94	1402	45	3.22	23.84	10010.227	25.0	16
2009	94	1402	50	3.22	23.82	10010.280	25.0	17

## APPENDIX E - THERMISTOR TEMPERATURE DERIVATION

Thermistor Type: YSI 44005, Dale #1C3001-B3, Alpha #13A3001-B3

Resistance to Temperature Equation:

$$T = \frac{1}{A + B(\ln R) + C(\ln R)^3} - 273.2$$

### Equation E-1 Convert Thermistor Resistance to Temperature

where: T = Temperature in °C.

LnR = Natural Log of Thermistor Resistance

A =  $1.4051 \times 10^{-3}$  (coefficients calculated over the -50 to +150° C. span)

B =  $2.369 \times 10^{-4}$

C =  $1.019 \times 10^{-7}$

Ohms	Temp	Ohms	Temp	Ohms	Temp	Ohms	Temp	Ohms	Temp
201.1K	-50	16.60K	-10	2417	+30	525.4	+70	153.2	+110
187.3K	-49	15.72K	-9	2317	31	507.8	71	149.0	111
174.5K	-48	14.90K	-8	2221	32	490.9	72	145.0	112
162.7K	-47	14.12K	-7	2130	33	474.7	73	141.1	113
151.7K	-46	13.39K	-6	2042	34	459.0	74	137.2	114
141.6K	-45	12.70K	-5	1959	35	444.0	75	133.6	115
132.2K	-44	12.05K	-4	1880	36	429.5	76	130.0	116
123.5K	-43	11.44K	-3	1805	37	415.6	77	126.5	117
115.4K	-42	10.86K	-2	1733	38	402.2	78	123.2	118
107.9K	-41	10.31K	-1	1664	39	389.3	79	119.9	119
101.0K	-40	9796	0	1598	40	376.9	80	116.8	120
94.48K	-39	9310	+1	1535	41	364.9	81	113.8	121
88.46K	-38	8851	2	1475	42	353.4	82	110.8	122
82.87K	-37	8417	3	1418	43	342.2	83	107.9	123
77.66K	-36	8006	4	1363	44	331.5	84	105.2	124
72.81K	-35	7618	5	1310	45	321.2	85	102.5	125
68.30K	-34	7252	6	1260	46	311.3	86	99.9	126
64.09K	-33	6905	7	1212	47	301.7	87	97.3	127
60.17K	-32	6576	8	1167	48	292.4	88	94.9	128
56.51K	-31	6265	9	1123	49	283.5	89	92.5	129
53.10K	-30	5971	10	1081	50	274.9	90	90.2	130
49.91K	-29	5692	11	1040	51	266.6	91	87.9	131
46.94K	-28	5427	12	1002	52	258.6	92	85.7	132
44.16K	-27	5177	13	965.0	53	250.9	93	83.6	133
41.56K	-26	4939	14	929.6	54	243.4	94	81.6	134
39.13K	-25	4714	15	895.8	55	236.2	95	79.6	135
36.86K	-24	4500	16	863.3	56	229.3	96	77.6	136
34.73K	-23	4297	17	832.2	57	222.6	97	75.8	137
32.74K	-22	4105	18	802.3	58	216.1	98	73.9	138
30.87K	-21	3922	19	773.7	59	209.8	99	72.2	139
29.13K	-20	3748	20	746.3	60	203.8	100	70.4	140
27.49K	-19	3583	21	719.9	61	197.9	101	68.8	141
25.95K	-18	3426	22	694.7	62	192.2	102	67.1	142
24.51K	-17	3277	23	670.4	63	186.8	103	65.5	143
23.16K	-16	3135	24	647.1	64	181.5	104	64.0	144
21.89K	-15	<b>3000</b>	<b>25</b>	624.7	65	176.4	105	62.5	145
20.70K	-14	2872	26	603.3	66	171.4	106	61.1	146
19.58K	-13	2750	27	582.6	67	166.7	107	59.6	147
18.52K	-12	2633	28	562.8	68	162.0	108	58.3	148
17.53K	-11	2523	29	543.7	69	157.6	109	56.8	149
								55.6	150

Table E-1 Thermistor Resistance versus Temperature

## **APPENDIX F - NETWORKING**

### **F.1. Description**

The Model LC-2 Datalogger is capable of being networked by way of a single, optically-isolated RS-485 communications cable. Utilizing one 8001-5 (RS-232) or 8002-5(USB) RS-485 interface adapter at the computer (data collection) end, up to 256 Model LC-2 Dataloggers\* may be networked. Also, the maximum network length\* can be up to 4000 feet (1.22 km). RS-485 is chosen as the transmission medium due to its inherent noise immunity and its capability to support a bus type of network architecture. The 8001-5 and 8002-5 RS-485 interface adapters are battery powered to allow for collection of data in the field. An AC adapter is also provided if mains power is available.

Each datalogger appears as a “node” on the RS-485 bus, with its own unique address. In order to communicate with a specific datalogger, the user transmits the address of the datalogger via the #nnn command, where nnn represents the network address of the datalogger. Valid addresses are 1 thru 256.

Connect one end of the supplied RS-485 network cable into the 10-pin “Network In” connector on the LC-2 enclosure, and connect the other end to the 10-pin “Network Out” connector on the 8001-5 or 8002-5 RS-485 interface. Connect additional LC-2 dataloggers in daisychain fashion, using RS-485 network cables to connect the “Network Out” of the first LC-2 to “Network In” of the second LC-2, “Network Out” of the second LC-2 to “Network In” of the third LC-2 and so on.

**On the last networked LC-2 (the one physically furthest from the 8001-5 or 8002-5 RS-485 interface), set the TERMINATION JUMPER across pins 1 and 2.** Depending on the LC-2 circuit board used, this jumper will be located either on a “daughterboard” mounted on top of the main circuit board – designated JP1, or on the main circuit board itself – designated JP-2. On all the remaining networked LC-2 dataloggers, ensure that this jumper is set across pins 2 and 3.

In order to access this jumper, the battery pack will need to be removed. For this reason, LC-2 networks are typically configured at the factory before shipment.

Finally, it is helpful to set the datalogger ID# (see section 4.9) to agree with the network address. This will tend to eliminate any confusion when collecting data.

For further information, refer to sections 4.9(ID), 4.20(NA), 4.21(NAddd), 4.22(ND), 4.23(NE) & 4.24(NS).

\* The total number of networked dataloggers is limited by the total network cable length. Contact a Geokon Sales Engineer for further information.



## F.2. Example of a 4 Datalogger Networking Session

1. This session assumes that there are 4 dataloggers running at 5 second scan intervals.
2. Press <ENTER> to wake the dataloggers from sleep. At this point, each datalogger is “listening” for its network address to be transmitted down the RS-485 bus.
3. To communicate with Datalogger #1 and observe several readings, type #1<ENTER>. Datalogger #1 returns:

Network address: 1

\*

1,2009,3,9,16,25,0,2.98,24.6,14.8114,20.5,1059

1,2009,3,9,16,25,5,2.98,24.7,14.8114,20.4,1060

1,2009,3,9,16,25,10,2.98,24.7,14.8114,20.5,1061

\*E

Note that the datalogger ID, which is the first entry for each ASCII character string, corresponds to the network address. **This should be set by the user during initial datalogger setup via the ID command.**

Typing E<ENTER> puts the datalogger back to sleep and disconnects it from the RS-485 bus. The datalogger will continue to wake up periodically (scan rate setting) to take a data reading. **The E command must be used in order to disconnect from the current datalogger and allow connection to the next datalogger.**

4. To communicate with Datalogger #2 and observe several readings, type <ENTER> to wake the dataloggers and then type #2<ENTER>. Datalogger #2 returns:

Network address: 2

\*

2,2009,3,9,16,23,25,2.95,24.7,14.8009,20.4,1040

2,2009,3,9,16,23,30,2.96,24.7,14.8009,20.4,1041

\*E

5. Doing the same for Datalogger numbers 3 & 4 results in:

Network address: 3

\*

3,2009,3,9,16,30,0,2.98,24.7,14.8116,20.5,1102

3,2009,3,9,16,30,5,2.98,24.7,14.8114,20.5,1103

\*E

Network address: 4

\*

4,2009,3,9,16,31,26,2.96,24.8,14.8110,20.4,1115

4,2009,3,9,16,31,31,2.96,24.8,14.8111,20.4,1116

\*E

## **APPENDIX G – Logware Compatibility**

### **G.1. Battery Gauge:**

The battery gauge display in Logware is inaccurate when used with the LC-2 datalogger and alkaline D-cell batteries.

Refer to the table below for the actual remaining battery life:

<b>Battery Voltage Display</b>	<b>Actual % Life Remaining</b>	<b>Logware Battery Gauge Display</b>
3.2	100	100
3.0	100	85.71
2.68	90	62.85
2.52	80	51.43
2.45	70	46.43
2.38	60	41.43
2.31	50	36.43
2.26	40	32.86
2.20	30	28.57
2.12	20	22.86
2.00	10	14.29
1.60	0	0

**Table G-1 Actual Battery Life vs. Logware Battery Gauge**

## **APPENDIX H – Lithium Coin Cell**

### **H.1. Description:**

Under normal operating conditions, the 1.5V ‘D’ cells provide all the power required to operate the LC-2 datalogger. In order to maintain the correct date and time settings for those periods when the ‘D’ cells are removed, the LC-2 datalogger incorporates a 3V lithium coin cell (Panasonic CR2032) to supply operating current to the internal Real Time Clock.

Since the power requirements of the Real Time Clock circuit are minimal (3 $\mu$ A max.), the clock will continue to operate for up to 10 years under these conditions.

However, if the lithium cell voltage falls to 2.5V or less, it should be replaced using the following replacement procedure.

### **H.2. Replacement Procedure:**

#### **Materials Required:**

1/4” Slotted Screwdriver  
1/8” Slotted Screwdriver  
1/4” Nutdriver  
CR2032 Lithium Coin Cell (Geokon P/N BAT-115)  
Disposable Grounding Wrist Strap (3M P/N 2209 or equivalent)

#### **Procedure:**

1. Put on the disposable grounding wrist strap and connect to a good earth ground.
2. Using the 1/4” slotted screwdriver, loosen the (4) captive screws and remove the datalogger cover.
3. Remove the two ‘D’ cells.
4. 8002-1-1, 8002-2-1, 8002-3-1: Using the 1/8” slotted screwdriver, disconnect the transducer wires at the terminal strip mounted on the battery plate.
5. Using the 1/4” slotted screwdriver, remove the (4) 3/8” 6x32 battery plate mounting screws.
6. Lift up the battery plate and disconnect both the 2 wire Molex connector from the header labeled “3V” (“12V” if applicable) and the transducer ribbon cable connector from the printed circuit board. Set the battery plate aside.
7. Using the 1/4” nutdriver, remove the (4) standoffs securing the printed circuit board to the case.
8. Lift the printed circuit board up to expose the bottom of the circuit board.
9. Using the 1/8” slotted screwdriver, gently pry the lithium coin cell battery from the battery holder.

10. Insert the replacement lithium coin cell into the battery holder (+ side facing out).
11. Re-install the printed circuit board back into the case.
12. Thread the (4) standoffs onto the set screws, using the nutdriver to gently tighten the standoffs.
13. Reconnect the transducer ribbon cable connector to the printed circuit board.
14. Reconnect the 2 wire Molex connector to the header labeled “3V” (“12V” if applicable).
15. Position the battery plate over the standoffs and re-install using the (4) 3/8” 6x32 battery plate mounting screws.
16. 8002-1-1, 8002-2-1, 8002-3-1:

Wire the terminal strip:

RED	VW+
BLACK	VW-
GREEN	TH+
WHITE	TH-
SHIELD	S

17. Re-install the ‘D’ cells.
18. Re-install the datalogger cover.

Lithium coin cell replacement complete.

**There's nothing more beautiful  
than a hard-working pump.**



**DEPENDABLE • LIGHT WEIGHT • EASY TO USE**

**12 Volt Submersible Pumps & Accessories**

**For Low Flow Sampling & Ground Water Purging**



**Proactive**  
**ENVIRONMENTAL PRODUCTS**

# Helping you get the job done right the first time!

Dependable – Lightweight- Easy to use 12 volt Groundwater pumps.

Building on over 18 years in business, Proactive Environmental Products understands your needs for reliable, accurate, and efficient groundwater pumps. Proactive anticipates the changing regulations in the groundwater sampling industry; therefore, continually improving our products based on our customers changing needs. This is the reason why we are the world’s premier manufacturer of 12 volt groundwater pumps and accessories with the reputation of simplifying groundwater sampling.

By means of innovation and quality control with every product we manufacture, rest assured when using a Proactive pump on your next groundwater sampling event, not only will you cut your sampling time in the field and increase your sampling accuracy, you will find it to truly be a more enjoyable experience.

This is our promise to you!

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## INDEX

Engineered Plastic Pumps - Standard . . . . .	1
Engineered Plastic Pumps - High Performance . .	4
Engineered Plastic Pumps Water Spout . . . . .	6
Stainless Steel Pumps . . . . .	7
Controllers and Power Boosters . . . . .	11
12 Volt Batteries . . . . .	14
Pump Charts . . . . .	16



## Simple to use. Hooks up to a 12 volt battery.

- When used in water, all pumps are rated for continuous use. No need for a cool down!
- All Pumps can run dry for up to 2 hours without motor damage!
- All pumps are auto-primed.
- All Pumps fit into a 2" well (Except the Abyss Pump).
- All Pumps are rated for over 400 hours of use when used in water.
- All pumps can be used in harsh conditions with higher turbidity.\*\*

**All 1.82" O.D. Engineered Plastic Pumps now feature a domed top and bottom. \***

Domed top minimizes well hang ups.

Fits 3/8" ID polyethylene tubing.

Reinforced, super strong discharge outlet (durable, field-tested).

Fits 1/2" ID polyethylene tubing.

Computer engineered domed bottom debris Screen will separate Debris from water, thus extending motor life.

## Non-Return (Non Vented) Check Valve

Top-fitting accessory used to prevent water back-flow when technician shuts off pump. Ideal for Low Flow Sampling.

Used With Standard and High Performance Engineered Plastic Pumps.

Item #PA-15020

## Accessory Item



# 12 VOLT STANDARD ENGINEERED PLASTIC PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging  
To Use, Just Hook Up To A 12 Volt Battery, Sound Simple? We Think So.*

## CYCLONE

**PUMPING DEPTH: 25 Ft. DTW\*** (7.62 Meters)

- Supplied with 35 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D.x7" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10100

## MINI-TYPHOON

**PUMPING DEPTH: 40ft. DTW\*** (12.2 Meters)

- Supplied with 50 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D.x12" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10150

## TYPHOON

**PUMPING DEPTH: 50ft. DTW\*** (15.25 Meters)

- Supplied with 60 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D.x12" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10200

## TEMPEST / TWISTER

**PUMPING DEPTH: 60ft. DTW\*** (18.3 Meters)

- Supplied with 70 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D.x17" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10250



# 12 VOLT STANDARD ENGINEERED PLASTIC PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging  
To Use, Just Hook Up To A 12 Volt Battery, Sound Simple? We Think So.*

## MINI-MONSOON



Item No. P-10300

**PUMPING DEPTH: 70ft. DTW\***  
(21.3 Meters)

- Supplied with 80 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 20" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.

## TORNADO



Item No. P-10330

**PUMPING DEPTH: 85ft. DTW\***  
(25 Meters)

- Supplied with 90 feet of heavy duty 10 gauge wire.
- Dimensions: 1.82" O.D. x 24" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.

## ABYSS

**3000 Hour  
Motor Life!**



Item No. P-10380

**PUMPING DEPTH: 220ft. DTW\***  
(67 Meters)

- Supplied with 230 feet of heavy duty 10 gauge wire.
- Dimensions: 3.5" O.D. x 12" Length.
- Use With: Optional Low Flow Sampling Controller (PA-10800).
- Use only 1/2" ID Low Density Polyethylene Tubing.
- Field replaceable wear out parts.
- 4 amp max consumption.
- Great for long term pump tests.

# 12 VOLT HIGH PERFORMANCE ENGINEERED PLASTIC PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging*

*To Use, Just Hook Up To A 12 Volt Battery, Sound Simple? We Think So.*

## MEGA-TYPHOON

### **PUMPING DEPTH: 80 Ft. DTW\***

(24.4 Meters)

- Supplied with 90 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 12" Length
- Requires: Power Booster 1 (PA-10600) or Low Flow with Power Booster 1 Controller (PA-10650)
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10350

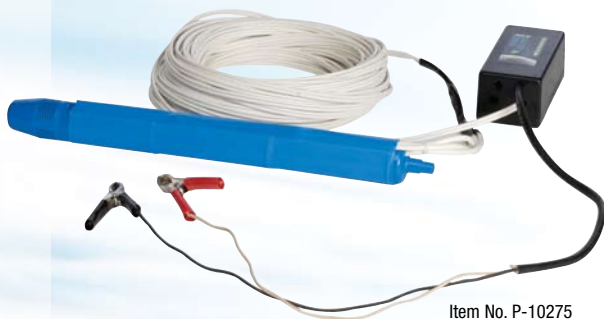
## SUPER TWISTER

### **PUMPING DEPTH: 85 Ft. DTW\***

(25 Meters)

- Supplied with 90 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 17" Length
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.
- No Power booster or controller needed to use this pump.
- Incorporates built in Booster Box.

*Not Compatible with any of  
ProActive's Controllers*



Item No. P-10275

## TSUNAMI

### **PUMPING DEPTH: 100 Ft. DTW\***

(30.5 Meters)

- Supplied with 110 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 17" Length
- Requires: Power Booster II (PA-10700) or Low Flow with Power Booster II (PA-10750)
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10400

*Perfect for Low Flow Sampling and Groundwater Purging  
To Use, Just Hook Up To A 12 Volt Battery, Sound Simple? We Think So.*

## MONSOON

**PUMPING DEPTH: 120 Ft. DTW\*** (37 Meters)

- Supplied with 130 feet of heavy duty 10 gauge wire.
- Dimensions: 1.82" O.D.x22" Length
- Requires: Power Booster II (PA-10700) or Low Flow with Power Booster II (PA-10750).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10500

## HURRICANE

**PUMPING DEPTH: 150ft. DTW\*** (46 Meters)

- Supplied with 160 feet of heavy duty 10 gauge wire and high impact plastic reel.
- Dimensions: 1.82" O.D.x26" Length.
- Requires: Power Booster II (PA 10700) or Low Flow with Power Booster II (PA-10750).
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



Item No. P-10510

## MEGA-MONSOON

**PUMPING DEPTH: 200ft. DTW\*** (61 Meters)

- Supplied with 210 feet of heavy duty 10 gauge wire.
- Dimensions: 1.82" O.D.x26" Length.
- Requires: Low Flow with Power Booster 3 (PA-10850).
- High Impact Plastic Reel Included.
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.



**IMPROVED DESIGN!  
MORE COMPACT,  
SAME POWER!**

Item No. P-10520

# 12 VOLT WATER SPOUT ENGINEERED PLASTIC PUMPS

*Perfect for Groundwater Purging*

## WATER SPOUT-1

SINGLE INLINE TOP PUMP

**PUMPING DEPTH: N/A**

- Supplied with 3 feet of wire.
- Dimensions: 1.82" O.D. x 7" Length.
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.
- Now you can build your own pump.

*NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS*



Item No. P-10000

## WATER SPOUT-1

DOUBLE BOTTOM BOOSTER PUMPS

**PUMPING DEPTH: 40 Ft. DTW\*** (12.2 Meters)

- Supplied with 3 feet of wire.
- Dimensions: 1.82" O.D. x 12" Length.
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.
- Now you can build your own pump.

*NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS.*



Item No. P-10010

## WATER SPOUT-1

COMPLETE

**PUMPING DEPTH: 40 Ft. DTW\*** (12.2 Meters)

- Supplied with 50 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 12" Length.
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.

*NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS.*



Item No. P-10020

## WATER SPOUT-2

COMPLETE

**PUMPING DEPTH: 60 Ft. DTW\*** (17 Meters)

- Supplied with 70 feet of heavy duty 12 gauge wire.
- Dimensions: 1.82" O.D. x 20" Length.
- Can be used with 3/8" or 1/2" ID Low Density Polyethylene Tubing.

*NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS.*



Item No. P-10050



# 12 VOLT STAINLESS STEEL PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging*

## Benefits of the Proactive Portable Stainless Steel Pump System

- **Simple to use! Runs off a 12 volt DC Battery.**
- Reliable Design is suitable for continuous sampling or purging of groundwater wells.
- Low Flow Sample as low as 10ML per minute when using Low Flow with proper Power Booster LCD 1,2, or 2.5 LCD controller & disposable Low Flow Control Valve.
- Long lasting, high performance pump motor lasts over 400 hours under normal working conditions.
- High polished stainless steel pump makes decontamination a breeze.
- Computer engineered bottom debris filter screen allows you to use pump in harsh conditions with higher turbidity.

## SET UP IN 60 SECONDS!

### Your Proactive Stainless Steel Pump System

*Pump, Controller and Battery Sold Separately.*



# 12 VOLT STAINLESS STEEL PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging*

## The Replaceable Stainless Steel Motor Module

*Designed for all Stainless Steel Pumps!*

### Only One Main Wear Out Part!

The main wear out parts consisting of the motor and seal all have been incorporated in a single replaceable motor module. Should the existing replaceable motor module fail in the field, a new replaceable motor module can quickly be installed within 60 seconds by a simple quarter turn twist! Once installed, your pump is essentially new and will have a life span identical to a new pump.



Quick change out motor module



Top Motor Housing Connector

## If you haven't used a Proactive Pump yet, you're missing out!

Have you had enough of dealing with a heavy, bulky and unreliable pump?

Now with a Proactive Pump, you can simplify your life!

- **Light Weight and Portable**
- **12 Volt Battery Powered**
- **Great for Low Flow Sampling and Ground Water Purging**
- **Simple Set Up**



Proactive pumps are so simple to set up! It can be done in 60 Seconds! It runs clean so you can eliminate the smelly fumes emitted by gas generators and potential air born cross-contamination. So, if you haven't tried one already, now is the time! Save your money, save your time and save your back, with a reliable Proactive Stainless Steel Portable 12 volt Pump.

# 12 VOLT STAINLESS STEEL PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging*

## SS MINI-MONSOON

- Great Substitute for Engineered Plastic Pumps.
- Easy to Decontaminate.
- Great for groundwater purging or long-term remediation projects.



**PUMPING DEPTH:**  
**80ft. DTW\***  
(24.4 Meters)

Dimensions: 1.82" O.D. x 7.5" Length.  
Item No. PS-10405

Supplied with

- 90 feet of heavy duty 10 gauge wire.
- 1 Motor Module Included on Pump Head.
- 1 Wrench.
- SS Mini-Monsoon Does Not Require a Power Booster or Controller.
- PUMP DOES NOT COME ON A REEL.

## SS MEGA TYPHOON

CONTROLLER SOLD SEPARATELY.



Easy To Read  
Voltage Output.

Dial Adjust Flow Control.  
Sound Simple? We Think So.

**PUMPING DEPTH:**  
**80ft. DTW\***  
(24.4 Meters)

Dimensions:  
1.82" O.D. x 6.5" Length.  
Item No. PS-10350

Supplied with

- 90 feet of heavy duty 12 gauge wire (Teflon Wire Upgrade Available).
- 1 Free Additional Motor Module.
- 1 Wrench.
- Disposable Low Flow Control Valve Included.
- Requires: Low Flow with Power Booster 1 "LCD" Controller (PA-10660)

Patent pending on all Stainless Steel Pumps with associated controllers.  
DTW Means Depth-To-Water. Specifications subject to change without notice.



# 12 VOLT STAINLESS STEEL PUMPS

*Perfect for Low Flow Sampling and Groundwater Purging*

## SS MONSOON

CONTROLLER SOLD SEPARATELY.

Easy To Read Voltage Output



Dial Adjust Flow Control  
Sound Simple? We Think So.

**PUMPING DEPTH:**  
**120 Ft. DTW\***  
(37 Meters)

Dimensions:  
1.82" O.D. x 7.5" Length.

Item No. PS-10400

Supplied with

- 130 feet of heavy duty 12 gauge wire. (Teflon Wire Upgrade Available).
- 1 Wrench.
- Disposable Low Flow Control Valve Included.
- 1 Free Additional Motor Module.
- Requires: Low Flow with Power Booster 2 "LCD" Controller (PA-10670).

## SS HURRICANE

CONTROLLER SOLD SEPARATELY.

Easy To Read Voltage Output



Dial Adjust Flow Control  
Sound Simple? We Think So.

**PUMPING DEPTH:**  
**150 Ft. DTW\***  
(45.7 Meters)

Dimensions:  
1.82" O.D. x 7.5" Length .

Item No. PS-10415

Supplied with

- 160 feet of heavy duty 10 gauge wire.
- 1 Wrench.
- Disposable Low Flow Control Valve Included.
- 1 Free Additional Motor Module.
- Requires: Low Flow with Power Booster 2.5 "LCD" Controller (PA-10675).

DTW Means Depth-To-Water.

Patent pending on all Stainless Steel Pumps with associated controllers. Specifications subject to change without notice.



# CONTROLLERS AND POWER BOOSTERS FOR ENGINEERED PLASTIC PUMPS

*Excluding Water Spout Line*

## POWER BOOSTER 2 CONTROLLER

### NOT SUITABLE FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use With the Engineered Plastic Tsunami, Monsoon and Hurricane.
- Easy access External Fuse.
- Will not turn pump motors off.



Item No. PA-10700

## LOW FLOW WITH POWER BOOSTER 2 CONTROLLER

### PERFECT FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use With the Engineered Plastic Tsunami, Monsoon and Hurricane.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.



Item No. PA-10750

## LOW FLOW WITH POWER BOOSTER 3 CONTROLLER

### PERFECT FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use With the Engineered Plastic Mega-Monsoon.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.



Item No. PA-10850

*Excluding Water Spout Line*

## LOW FLOW SAMPLING CONTROLLER



Cigarette Lighter Adaptor



### PERFECT FOR LOW FLOW SAMPLING.

- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates thermal overload protection.
- Use with proactive's complete standard pump line.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.

Item No. PA-10800

## POWER BOOSTER 1 CONTROLLER



### NOT SUITABLE FOR LOW FLOW SAMPLING.

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use With the Engineered Plastic Mega-Typhoon.
- Easy access External Fuse.
- Will not turn pump motors off.

Item No. PA-10600

## LOW FLOW WITH POWER BOOSTER 1 CONTROLLER



### PERFECT FOR LOW FLOW SAMPLING.

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use With the Engineered Plastic Mega-Typhoon.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.

Item No. PA-10650

## LOW FLOW WITH POWER BOOSTER 1 "LCD" CONTROLLER



### PERFECT FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use with the SS-Mega-Typhoon.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.

## LOW FLOW WITH POWER BOOSTER 2 "LCD" CONTROLLER



### PERFECT FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use with the SS-Monsoon.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.

## LOW FLOW WITH POWER BOOSTER 2.5 "LCD" CONTROLLER



### PERFECT FOR LOW FLOW SAMPLING

- Consistent Voltage Regulation Technology (CVRT).
- Controller is engineered with a reverse polarity protection circuitry.
- Incorporates a current limitation circuitry and thermal overload protection.
- Incorporates a low voltage disconnect circuitry (LVD).
- Use with the SS-Hurricane.
- Easy access External Fuse.
- Low flow sample down to 10ml per minute with controller and disposable low flow control valve.
- Able to turn pump motors off.

# MOBILE BATTERY POWER PACK

## 38 Amp Extended Life

Proactive 38 Amp Mobile Battery Power Packs is a compact, durable and portable 12 volt system specifically designed for Proactive's Standard and Water Spout pump line. This self-contained, rechargeable 12 volt system can very easily replace gas generators or your old car battery.

### Cigarette Lighter Plug - Great for ProActive Pumps!

- Light Weight - Only 38 pounds.
- Heavy duty polyethylene construction.
- Convenient handle for easy portability.
- Reverse polarity protection.
- Overcharge protection.
- Built in light for night time sampling.
- AC or DC recharging options.
- Fuse Protected.
- Convenient On/Off Switch.



Item No. PB-20010

**100% longer lasting than a standard 12 volt car battery.**

## 12 Volt Proactive AGM Batteries

Our 12 volt AGM non spillable Batteries are a totally sealed and maintenance free design. There are no discharge tubes or fillers caps, which eliminates the need to maintain water levels and offers no concern about acid leaks on valuable parts and accessories.

Batteries are able to ship Federal Express air or Federal Express ground\*\*. Slower self discharge rate (longer shelf life) means that these batteries can sit for extended periods of time without constant monitoring. A wet battery (car battery) discharges 15% a month, where our AGM batteries discharge only 2-3% a month. All our AGM Batteries are backed by a 6 month warranty.

## 38 AMP BATTERY

### AGM Non Spillable Battery

- Length: 7.69 in • Width: 6.50 in
- Height: 7.19 in • Weight: 29 lbs

**100% longer lasting than a standard 12 volt car battery.**

*Does not come with carrying handle!*



Item No. PB-16070





Item No. PB-16080

## 55 AMP BATTERY

### AGM Non Spillable Battery

Carrying Handle for easy transport.

- Length: 9.0 in • Width: 5.44 in
- Height: 9.0 in • Weight: 39 lbs

**200% longer lasting than a standard 12 volt car battery**



Item No. PB-16090

## 75 AMP BATTERY

### AGM Non Spillable Battery

- Carrying Handle for easy transport.
- Length: 10.25 in • Width: 6.13 in
- Height: 9.13 in • Weight: 55 lbs

**310% longer lasting than a standard 12 volt car battery**



Item No. PB-16100

## 90 AMP BATTERY

### AGM Non Spillable Battery

- Carrying Handle for easy transport.
- Length: 12.0 in • Width: 6.63 in
- Height: 9.06 in • Weight: 63.40 lbs

**400% longer lasting than a standard 12 volt car battery**

## 1 AMP BATTERY CHARGER



Item No. PB-16000

- Plug into 120 volt outlet to charge all Proactive's 12 volt batteries.
- 12 volt 1 amp regulated dual stage smart battery charger incorporates a charged and charging status light letting you know when the battery is fully charged.
- Specifically designed for lead acid batteries.
- Designed for a variety of applications.
- Connector: Color coded alligator clips.
- 6 month Warranty.

# 12 VOLT PUMP CHARTS

## STANDARD ENGINEERED PLASTIC PUMPS

### PUMPING DEPTH IN FEET

PUMP MODEL	ITEM NO.	OPTIONAL CONTROLLER	3	10	20	25	30	40	50	55	60	70	85	90
<b>CYCLONE</b> with 35' of wire	P-10100	LOW FLOW SAMPLING CONTROLLER (PA-10800)	2.0 G.P.M 12V.	1.0 G.P.M 12V.	.35 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
<b>MINI-TYPHOON</b> with 50' of wire	P-10150	LOW FLOW SAMPLING CONTROLLER (PA-10800)	2.5 G.P.M 12V.	2.2 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	1.0 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A
<b>TYPHOON</b> with 60' of wire	P-10200	LOW FLOW SAMPLING CONTROLLER (PA-10800)	3.0 G.P.M 12V.	2.3 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	1.0 G.P.M 12V.	.6 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A
<b>TEMPEST / TWISTER</b> with 70' of wire	P-10250	LOW FLOW SAMPLING CONTROLLER (PA-10800)	3.2 G.P.M 12V.	2.8 G.P.M 12V.	2.2 G.P.M 12V.	2.0 G.P.M 12V.	1.75 G.P.M 12V.	1.2 G.P.M 12V.	.75 G.P.M 12V.	.60 G.P.M 12V.	.40 G.P.M 12V.	N/A	N/A	N/A
<b>MINI-MONSOON</b> with 80' of wire	P-10300	LOW FLOW SAMPLING CONTROLLER (PA-10800)	4.0 G.P.M 12V.	3.2 G.P.M 12V.	2.8 G.P.M 12V.	2.5 G.P.M 12V.	2.2 G.P.M 12V.	1.5 G.P.M 12V.	1.0 G.P.M 12V.	.75 G.P.M 12V.	.60 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A
<b>TORNADO</b> with 90' of wire	P-10330	LOW FLOW SAMPLING CONTROLLER (PA-10800)	4.25 G.P.M 12V.	3.75 G.P.M 12V.	3.5 G.P.M 12V.	3.25 G.P.M 12V.	3.0 G.P.M 12V.	2.5 G.P.M 12V.	1.75 G.P.M 12V.	1.25 G.P.M 12V.	.75 G.P.M 12V.	.50 G.P.M 12V.	.35 G.P.M 12V.	N/A

PUMP MODEL	ITEM NO.	OPTIONAL CONTROLLER	10	20	40	60	80	100	120	140	160	180	200	220
<b>ABYSS</b> with 230' of wire	P-10380	LOW FLOW SAMPLING CONTROLLER (PA-10800)	1.15 G.P.M 12V.	1.0 G.P.M 12V.	1.0 G.P.M 12V.	.85 G.P.M 12V.	.85 G.P.M 12V.	.80 G.P.M 12V.	.80 G.P.M 12V.	.75 G.P.M 12V.	.75 G.P.M 12V.	.70 G.P.M 12V.	.70 G.P.M 12V.	.50 G.P.M 12V.

## WATER SPOUT ENGINEERED PLASTIC PUMPS

### PUMPING DEPTH IN FEET

PUMP MODEL	ITEM NO.	OPTIONAL CONTROLLER	3	10	20	25	30	40	50	55	60	70	85	90
<b>WATER SPOUT 1 TOP INLINE PUMP</b> 1-Stage, with 3' of wire*	P-10000	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
<b>WATER SPOUT 1 DOUBLE BOTTOM BOOSTER</b> 2-Stage, with 3' of wire*	P-10010	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
<b>WATER SPOUT 1 COMPLETE</b> with 50' of wire	P-10020	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	2.5 G.P.M 12V.	2.2 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	1.0 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A
<b>WATER SPOUT 2 COMPLETE</b> with 70' of wire	P-10050	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	3.2 G.P.M 12V.	2.75 G.P.M 12V.	2.5 G.P.M 12V.	2.25 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	.75 G.P.M 12V.	.50 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A

\*Pump cannot be used by itself.

Pump test results were conducted with a 12.5 volt source.

DTW means Depth-To-Water. Specifications subject to change without notice.

## HIGH PERFORMANCE ENGINEERED PLASTIC PUMPS

### PUMPING DEPTH IN FEET

PUMP MODEL	ITEM NO.	REQUIRED CONTROLLERS	10	20	40	50	70	80	85	100	110	120	140	150	180	200
<b>MEGA-TYPHOON</b> with 90' of wire	P-10350	POWER BOOSTER 1 (PA-10600) OR LOW FLOW WITH POWER BOOSTER 1 (PA-10650)	3.5 G.P.M 12V.	3.1 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	.5 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
<b>SUPER TWISTER</b> with 90' of wire	P-10275	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	2.5 G.P.M 12V.	2.0 G.P.M 12V.	1.50 G.P.M 12V.	1.25 G.P.M 12V.	.75 G.P.M 12V.	.50 G.P.M 12V.	.50 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A	N/A
<b>TSUNAMI</b> with 110' of wire	P-10400	POWER BOOSTER 2 (PA-10700) OR LOW FLOW WITH POWER BOOSTER 2 (PA-10750)	4.0 G.P.M 12V.	3.8 G.P.M 12V.	3.2 G.P.M 12V.	3.0 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	1.0 G.P.M 12V.	.50 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A	N/A
<b>MONSOON</b> with 130' of wire	P-10500	POWER BOOSTER 2 (PA-10700) OR LOW FLOW WITH POWER BOOSTER 2 (PA-10750)	4.4 G.P.M 12V.	4.2 G.P.M 12V.	3.5 G.P.M 12V.	2.8 G.P.M 12V.	2.1 G.P.M 12V.	1.9 G.P.M 12V.	1.6 G.P.M 12V.	1.5 G.P.M 12V.	1.25 G.P.M 12V.	1.0 G.P.M 12V.	N/A	N/A	N/A	N/A
<b>HURRICANE</b> with 160' of wire	P-10510	POWER BOOSTER 2 (PA-10700) OR LOW FLOW WITH POWER BOOSTER 2 (PA-10750)	4.5 G.P.M 12V.	4.0 G.P.M 12V.	3.0 G.P.M 12V.	2.75 G.P.M 12V.	2.25 G.P.M 12V.	2.0 G.P.M 12V.	1.75 G.P.M 12V.	1.35 G.P.M 12V.	1.25 G.P.M 12V.	1.15 G.P.M 12V.	.50 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A
<b>MEGA-MONSOON</b> with 210' of wire	P-10520	LOW FLOW WITH POWER BOOSTER 3 (PA-10850)	4.0 G.P.M 12V.	3.75 G.P.M 12V.	2.75 G.P.M 12V.	2.5 G.P.M 12V.	2.0 G.P.M 12V.	1.75 G.P.M 12V.	1.75 G.P.M 12V.	1.65 G.P.M 12V.	1.65 G.P.M 12V.	1.5 G.P.M 12V.	.65 G.P.M 12V.	.50 G.P.M 12V.	.30 G.P.M 12V.	.15 G.P.M 12V.

## STAINLESS STEEL (SS) PUMPS

### PUMPING DEPTH IN FEET

PUMP MODEL	ITEM NO.	REQUIRED CONTROLLER	10	20	30	40	60	70	80	100	110	120	130	150
<b>SS-MINI MONSOON</b> with 90' of wire	PS-10405	NOT COMPATIBLE WITH ANY OF PROACTIVE'S CONTROLLERS	1.75 G.P.M 12V.	1.25 G.P.M 12V.	1.25 G.P.M 12V.	1.0 G.P.M 12V.	.75 G.P.M 12V.	.50 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A
<b>SS-MEGA-TYPHOON</b> with 90' of wire	PS-10350	LOW FLOW WITH POWER BOOSTER 1 "LCD" CONTROLLER (PA-10660)	3.0 G.P.M 12V.	2.75 G.P.M 12V.	2.55 G.P.M 12V.	2.0 G.P.M 12V.	1.0 G.P.M 12V.	.50 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A	N/A	N/A	N/A
<b>SS-MONSOON</b> with 130' of wire	PS-10400	LOW FLOW WITH POWER BOOSTER 2 "LCD" CONTROLLER (PA-10670)	3.5 G.P.M 12V.	3.25 G.P.M 12V.	3.0 G.P.M 12V.	3.0 G.P.M 12V.	2.0 G.P.M 12V.	1.75 G.P.M 12V.	1.25 G.P.M 12V.	1.0 G.P.M 12V.	.50 G.P.M 12V.	.25 G.P.M 12V.	N/A	N/A
<b>SS-HURRICANE</b> with 160' of wire	PS-10415	LOW FLOW WITH POWER BOOSTER 2.5 "LCD" CONTROLLER (PA-10675)	3.5 G.P.M 12V.	3.25 G.P.M 12V.	3.0 G.P.M 12V.	3.0 G.P.M 12V.	2.5 G.P.M 12V.	2.0 G.P.M 12V.	1.5 G.P.M 12V.	1.25 G.P.M 12V.	1.25 G.P.M 12V.	1.0 G.P.M 12V.	.85 G.P.M 12V.	.50 G.P.M 12V.

Pump test results were conducted with a 12.5 volt source.

DTW means Depth-To-Water . Specifications subject to change without notice.



GROUNDWATER  
*Essentials*

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